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HISTORY OF THE QUARTZ OSCILLATOR-PLATE INDUSTRY, 1941–1944

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In 1881, Pierre and Jacques Curie discovered that quartz and certain other low symmetry crystals developed positive and negative electrical charges at the ends of the polar $X=a$-axes when subjected to properly directed pressure. In the following year, G. Lippmann suggested that such crystals would become mechanically deformed if subjected to an electrical field, and this converse piezoelectric effect, so-called, was experimentally verified by the Curies. The effect remained hardly more than a laboratory curiosity for many years until P. Langevin in Paris, during World War I, devised quartz plates for sending out and detecting underwater sound waves. At about the same time, A. M. Nicholson of the Bell Laboratories experimented with phonograph pickups and microphones constructed of Rochelle-salt crystals. The application of the piezoelectric effect to radio, however, did not come until 1921, when Professor W. G. Cady of Wesleyan University showed that quartz plates could be used to control vacuum tube oscillators. In this application, the alternating electrical field generated by the vacuum tube radio circuit is applied to a quartz plate, properly cut and mounted and so dimensioned that one of its natural frequencies of mechanical vibration coincides with the oscillations of the circuit, and the frequency of transmission or reception is thereby stabilized and precisely controlled. Some types of quartz oscillator-plates used by the Armed Services are shown in Fig. 1. Quartz, rather than other natural or artificial crystals, some of which give much more pronounced piezoelectric effects, is employed for the purpose primarily because of its chemical and physical stability, high elasticity and availability. An increasing demand for crystal-controlled oscillators and filter circuits developed during the 1930's and approximately 50,000 quartz oscillator-plates were manufactured in the United States during 1939.

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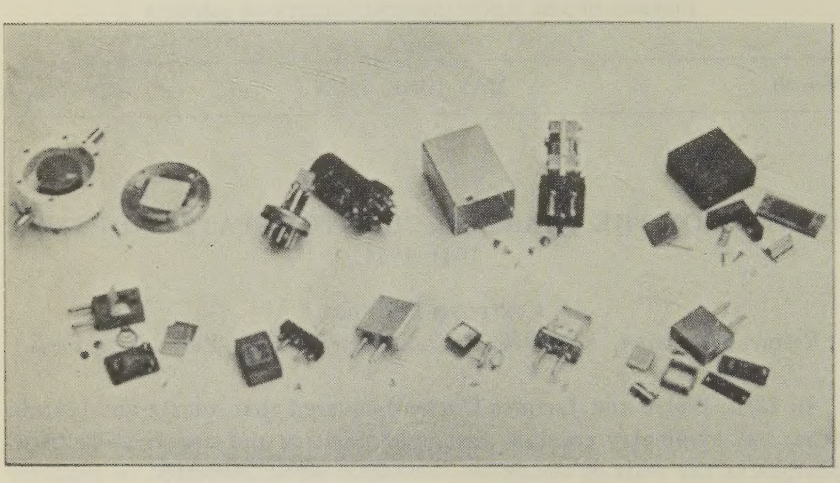

FIG. 1. Some types of mounted quartz oscillator-plates for radio use. No. 1, FT-243 type clamped crystal, showing component parts. No. 2, FT-241 wire suspension metal-plated CT-cut crystal. No. 3, hermetically sealed CR-1 crystal in solder-sealed all-metal holder. No. 4, CR-7 type, plated wire suspension BT-cut crystal. No. 5, hermetically sealed CR-1 type crystal in solder-sealed Pyrex glass holder. No. 6, CR-1 clamped type crystal in phenolic holder. No. 7, FT-164 AT-cut crystal in variable air gap holder. No. 8, metal-plated wire suspension DT-cut crystal. No. 9, MX-12 AT-cut clamped crystals in dual mounting. No. 10, DC-11 type clamped crystal, showing L-shaped adapter to accommodate small sized crystal within the holder.

With entrance of the United States into World War II in 1941 the Armed Services demanded almost fantastic quantities of oscillator plates —literally tens of millions. Part of the needs of our Allies also were to be supplied by Lend-Lease. Rapid and certain communication is the keystone of global war, and many thousands of aircraft, ships, tanks or other mobile or stationary ground units were to be fitted with radios, each with its complement of "crystals." The problem of supply created an industrial problem of the first magnitude. The responsibility for initiating and guiding procurement for the Army and Air Forces rested largely in the Quartz Crystal Coordination Section, General Development Division, Office of the Chief Signal Officer, War Department. This group was activated under a directive of March 5, 1942, from Major General (then Brigadier General) Roger B. Colton, Chief of the Material Branch, U. S. Army Signal Corps, which charged the General Development Division, among other duties, to "Take necessary steps to handle the crystal problem and successfully meet it." The Quartz Crystal Coordination Section had its headquarters in Temporary Building A, later in the Pentagon Building, Washington, and comprised a small number of civilian and Army technicians and scientists working under the direction originally of Colonel (then Lieutenant Colonel) J. D. O'Connell. This group carried the brunt of the work during the first year or so of the crystal program. The Crystal Section was aided in its task by the Crystal Branch of the Signal Corps Laboratories at Fort Monmouth, New Jersey, and later by the Aircraft Radio Laboratory at Wright Field, Ohio. The Fort Monmouth Crystal Laboratories (later located at Camp Coles and then at Long Branch, New Jersey) was active primarily in placing educational orders enabling manufacturers to obtain necessary priorities and get set up, in awarding Development Contracts on the design and development of equipment and to investigate technical problems, and in research work. The Laboratories also operated a Pilot Plant and for a brief period gave courses of instruction to new manufacturers. The present laboratory activity includes the development of all crystals used in ground force signal equipment. The Crystal Branch of the ARL, beginning in 1943, did valuable laboratory and field work and contributed to the increase in quality of both oscillator-plates and holders.

Special acknowledgement also should be made to the Bell Telephone Laboratories and the Western Electric Company, who contributed greatly to the success of the crystal program. These companies freely supplied invaluable technical information and led in the development of several types of manufacturing equipment, notably the drill press lap, immersion conoscope, inspection tank for raw quartz and x-ray methods for controlling orientation. The fundamental theory and design of oscillator-plates is largely owing to work carried on over many years at the Bell Telephone Laboratories. They developed low temperature coefficient crystals and associated electronic equipment, studied the modes of vibration of oscillator crystals and predimensioning, and developed wire suspension metal-plated crystals.

During 1942 and the early months of 1943 about 100 new manufacturers, in addition to a dozen or so older companies, were in the production field. Several received direct government financial aid. The Signal Corps furnished instruction to all plants needing it, and also equipped many plants. This industry, driven by insistent demands of the Armed Services for immediate and large production, from 1941 to the end of 1944 delivered roughly 55,000,000 quartz oscillator-plates. The total amount of raw quartz used to manufacture these crystals amounted to thousands of tons. In addition, probably half again as much quartz was partly or completely processed but was wasted or unusable due variously to ignorance, carelessness, haste and lack of adequate technical controls. The cost of tooling up and doing the job amounted to about a third of a billion dollars. Production still is continuing on a large scale. A tabulation of the principal types of oscillator-plates put into mass production from 1942 to date is given on the next page.

Some of the difficulties that had to be overcome in establishing the crystal industry may be mentioned. Great quantities of raw quartz crystals had to be supplied. This material was obtained entirely from Brazil, where the purchasing and initial grading was carried on under the direction of governmental agencies and private importers. A considerable effort also was made to extend and mechanize the primitive quartz mining and distributing industry in Brazil. A stockpile of some millions of pounds of quartz was built up in the United States by the Metals Reserve Corporation, the material being graded by the National Bureau of Standards, and this and privately imported quartz was allocated to industry by the Miscellaneous Minerals Section of the War Production Board. During 1942 the supply situation became acute due in part to difficulties of sea transport and considerable amounts of quartz were flown by plane from Brazil to the United States and even to England. Efforts by Government agencies and others to find or develop commercial supplies of raw quartz within the United States proved unsuccessful and only a few tons of domestic quartz, mostly from Arkansas, were processed. Unsuccessful attempts were made to synthesize large quartz crystals. The price of faced raw quartz crystals ranged from about $5 to $10 per pound for material of average quality and up to $30 and more with increasing size and perfection. Raw quartz crystals of average size and quality are shown in Fig. 2. One pound of quartz yielded on the average little more than a dozen BT-cut oscillator-plates. Unfaced quartz was relatively abundant and cheap but efforts made to equip and teach the industry to handle such material met with reluctance.

The problem of taking a prewar laboratory-scale quartz cutting industry and equipping it almost overnight on a mass production scale with modern precision tools was very great. For many types of equipment, especially lapping machines, *x*-ray goniometric apparatus and frequency measuring equipment, designs had to be created. Manufacturers were found or created to make the equipment and then supplied

TABLE 1. DELIVERY OF QUARTZ OSCILLATOR-PLATES FROM THE CRYSTAL INDUSTRY TO THE ARMED SERVICES, 1941–1945[1]

Type[3]	Remarks[2]	1941	1942	1943	1944	1945
FT-171-B	AT 1.5 to 6.0 mc. Freq. tol. = ±0.02%. Temp. range −40° to +70°C. Used in armored force, field troop and artillery command sets.		132,351	846,521	521,014	
FT-241-A	DT-cut, 200 Kc. CT-cut, 300 to 1200 Kc. Tol. = ±0.02%. Range −40° to +70°C. Tank and other ground vehicle radios; field artillery.		2,836,293	3,568,651	4,488,362	
FT-243	AT and BT 2.0 to 9.0 mc. Freq. tol. = ±0.02%. Range −40° to +70°C (varies). Walkie-talkie, handie-talkie, and other portable combat sets; field artillery sets.		2,104,712	9,711,967	11,000,469	
DC-8	AT and BT 4.3 to 9.0 mc. Range −30° to +50°C. Aircraft command sets.		122,681	370,702	141,552	
DC-9	AT 1000 Kc. 15 cycles at 20° and 60 cycles over −30° to +50°C; operative −40° to +70°C. Calibrator crystal in frequency meter.		33,979	117,655	28,520	
DC-11	BT 5 to 9 mc. Freq. tol. = ±0.02%. Range −55° to +90°C. (A grade) Aircraft radios.		214,370	1,860,499	536,802	
DC-20	BT 5.6 to 5.7 mc. Freq. tol. = ±0.02%. Range −40° to +85°C. Airforce; runway localizer.		34,561	235,751	32,745	
DC-26	BT 5 to 9 mc. Predecessor to CR-1. Aircraft radio.		—	787,755	5,139	
DC-34 DC-35	AT or BT 1.6 to 4.5 mc. Freq. tol. = ±0.02%. Range −40° to +70°C. Ground forces; vehicles.		—	42,941	1,770,999	
CR-1A/AR	BT 5 to 9 mc. Freq. tol. = ±0.02%. Range −55° to +90°C (A grade) Aircraft radios.		—	1,359,957	7,274,909	
Other[4]			372,525	1,260,215	2,783,474	
	Totals	100,000 (est.)	5,851,472	20,362,614	28,583,985	30,000,000 (est.)

[1] Figures furnished by the Signal Corps, include Lend-Lease shipments.

[2] Tolerances and temperature ranges are those currently used, and are considerably more stringent than those in effect on 1941, 1942 and most of 1943.

[3] FT numbers refer only to holders; DC and CR numbers refer to complete units.

[4] Includes Navy crystals, in part of above mentioned types.

with machine tools and other critical materials. A number of these operations were bolstered with high priorities and financed with Government funds from the Defense Supplies Corporation. The phrase "priming the pump" was popular in Washington at this time. Most of the equipment was held in Signal Corps pools and allocated to the industry. While not crystal manufacturers themselves, the Galvin Manufacturing Corporation of Chicago coordinated more than 30 subcontractors located over the entire country who made crystals in holder type FT-243 for "walkie-talkie" and other ground radio equipment of their manufacture. Galvin formed several equipment pools to supplement those formed by the Government, the most important item covered being the Atlas drill press lap, and among other services issued a series of short mimeographed bulletins (62 to date) dealing mainly with practical matters in crystal manufacturing. The North American Philips X-Ray Co., Inc., and later the General Electric X-Ray Corp. undertook the design and manufacture of x-ray equipment to control cutting angles and together have delivered

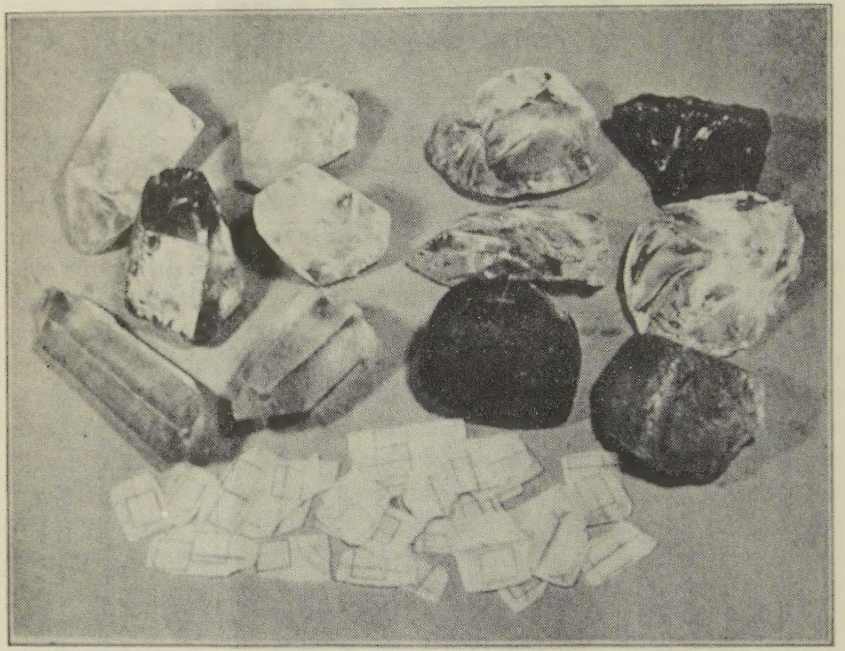

FIG. 2. Faced (left) and unfaced (right) masses of quartz of average size. The sawn wafers have 0.5×0.6 inch blanks marked out thereon.

about 300 units to the industry. The Naval Research Laboratory, Anacostia Station, D. C., developed the sensitive Geiger counter and electronic circuit for the Philips unit.

A number of crystal manufacturers also made important contributions to the art. The Bliley Electric Co., Bell Telephone Laboratories and Harvey-Wells Communication, Inc., pioneered the etching-to-frequency method of finishing crystals.[1] The Bendix Radio Corporation of Baltimore cooperated with Signal Corps technicians in the development of crystal cutting procedures, developed the parallelogram light-figure method of orientation, and contributed to the design of both crystal lapping and *x*-ray orientation equipment. The G. C. Hunt Co. and P. R. Hoffman Co., both of Carlisle, Penna., developed the precision planetary lap. The milling method of finishing crystals was developed in the laboratory of the Galvin Manufacturing Co., Chicago. The North American Philips Co., Dobbs Ferry, N. Y., provided facilities for studying the orientation and cutting procedures described in a series of official Signal Corps publications, developed precision lapping and cutting equipment and methods in their Pilot Plant, and contributed in various ways to *x*-ray methods of orienting quartz. The North American Philips X-Ray Co. also designed and constructed equipment for the *x*-ray irradiation method of adjusting crystal frequency. The Reeves Sound Laboratories, Inc., of New York City, discovered and applied the *x*-ray irradiation technique of adjusting crystal frequency, developed safe etching compounds for quartz, and helped investigate the cause and cure of crystal ageing.

Among the principal difficulties confronting the newly established industry were uninformed management, the scarcity of skilled personnel, and the general lack of technical information, especially that dealing with the practicalities of orienting quartz and cutting plates therefrom. The prewar experience of many of the new manufacturers was in such fields as the manufacture of lamp shades, grindstones, transformers, neon lamps, sound recording apparatus and butchers' supplies. One was an undertaker. More than one of these new companies soon were producing more crystals in a week than the whole country did in the year 1939. Electronic engineers with or without a knowledge of quartz oscillator-plates, crystal physicists and crystallographers were hard to find and desperately sought. Many potentially useful men had already been drafted into the Armed Services. The Signal Corps did its best to help this situation by field visits of staff technicians and by publication[2] of a series of mimeographed technical handbooks and bulletins written by members of the Quartz Crystal Coordination Section, Washington, and the Signal Corps Laboratories. Steps were taken, as in other critical industries, to prevent the drafting of key men. There was also a free and ready interchange of technical advice and assistance between the various manufacturers.

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[1] The method of etching quartz plates to frequency with hydrofluoric acid was patented (No. 1,869,160) in May, 1928, by W. A. Marrison of the Bell Telephone Laboratories.

Technical difficulties also beset the new industry. The so-called Beilby-

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[2] Publications of the Quartz Crystal Section, OCSigO, include:

Frondel, Clifford, and others, Handbook for the Manufacture of Quartz Oscillator-Plates, August 20, 1942.

The following six information bulletins were issued as supplements to the Handbook:

Waesche, Hugh H., Procedure for finishing quartz oscillators, Inf. Bull., No. 1, Sept. 25, 1942.

Waesche, Hugh H., Equipment used for quartz crystal finishing and testing, Inf. Bull., No. 2, Sept. 25, 1942.

Woods, E. K., Memorandums on ordering information, Inf. Bull., No. 3, Sept. 24, 1942.

Richmond, Wallace E., Technique of sawing quartz, Inf. Bull., No. 4, Oct. 12, 1942.

Waesche, Hugh H., and Wolfskill, John M., A method of orientation and sawing of small unfaced quartz, Inf. Bull., No. 5, March 1943.

Frondel, Clifford, Technical practices in the crystal industry of the United Kingdom, Inf. Bull., No. 5, 1943.

Parrish, William, and Gordon, Samuel G., Manual for the manufacture of quartz oscillator-blanks, February 15, 1943.

The following four papers were issued as supplements to this widely used Manual:

Parrish, William, and Gordon, Samuel G., Procedures for unfaced quartz and large quartz, Supp. No. 1, March 31, 1943.

Gordon, Samuel G., The inspection of quartz, Supp. No. 2. July 1, 1943.

Gordon, Samuel G., Salvage of quartz and reclamation of rejected crystals, Supp. No. 3, July 1, 1943.

Bottom, Virgil E., Procedures for etching crystals to frequency, Supp. No. 4, Aug. 1, 1944.

Publications of the Fort Monmouth (now Long Branch) Crystal Laboratories (Signal Corps Ground Signal Agency) comprise 21 articles to date, among which may be mentioned:

Technical News Bulletin #5, "Orientation Method for the Use of Small Unfaced Quartz," 1942.

Technical News Bulletin #7, "Limits of Usability of Raw Quartz," 1943.

Technical News Bulletin #9, "Procedure for Cleaning Crystal Blanks," September 1943.

Engineering Memo. #1, September 1943, "Recommended Procedure for Predimensioning Broad Frequency Tolerance AT and BT Oscillator-Plates."

Engineering Report #778, January 1943, "*X*-Ray Orientation of Small Unfaced Quartz."

Engineering Memo. #4, June 1944, "Ageing of Quartz Crystal Units."

Engineering Report #12, August 1943, "Small DC-9 Crystals."

Engineering Report #7, April 1943, "The Impedance of FT-243 Crystals."

layer, debated pro and con for many years, turned out to be an unfortunate reality that in 1943 nearly caused cessation of manufacture of high frequency crystals. It was found that millions of crystals held in storage in Signal Corps depots or issued to the Services had deteriorated to such extent as to make them unusable. The ageing phenomenon commonly appeared as a spontaneous increase in frequency with an accompanying partial or complete loss of "activity." The effect was traced to changes with time in a thin skin of misaligned and possibly hydrated quartz produced by abrasive action on the plate during manufacture. Etching in a solvent to remove this layer was found to obviate the effect. It was also found that corrosion and water absorption occurred to an objectionable degree in phenolic type crystal holders when used in the tropics, and this led to the use of improved plastics relatively impervious to moisture and to the accelerated development, still under way, of hermetically sealed glass or all-metal holders. Predimensioning became the subject of much concern. Predimensioned crystals are cut to certain rigidly specified predetermined physical dimensions that ensure satisfactory performance, but most manufacturers preferred for various reasons, especially in high frequency plates, to cut to arbitrary dimensions and hand tailor each crystal individually by edge lapping to give maximum performance. The Signal Corps exerted pressure on the industry to increase the efficiency and economy of manufacture and the quality of the product. Outstanding production records in certain plants and price renegotiation also acted to stimulate the industry in these regards. During 1943 special efforts were made to conserve quartz by improving cutting methods and reducing crystal size. The use of a proportion of unfaced quartz was made obligatory on most contracts. Increasing emphasis was placed on care in finishing the crystal, cleanliness and final inspection. In 1944, etching-to-frequency techniques were made mandatory, and the specifications of crystals with regard to temperature testing range and frequency and activity tolerances were further tightened. More recently, hermetically sealed holders are required for certain types of crystals in place of the older phenolic types. Price competition—largely lacking in preceding years of huge demand and small supply—is becoming marked.

The following Symposium of papers is primarily concerned with descriptions of the occurrence and characters of industrial quartz crystals and of the orientation methods used in manufacture. The principal manufacturing methods and tools also are described in some detail, but the electronic side of the field is touched only in passing. The manufacturing methods described are principally those used in the mass production of shear mode BT and AT plates.

THE PIEZOELECTRIC QUARTZ RESONATOR

KARL S. VAN DYKE*

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I. THE CONTRIBUTING PROPERTIES OF QUARTZ

A. *Highly Perfect Elastic Properties*

The excellence of quartz for use in frequency control is expressed in the large numerical values of Q in quartz resonators, which the use of this highly elastic material makes possible. Q is a number used in acoustics and in electrical engineering in listing, for vibrating systems, their relative efficiencies of transfer of energy back and forth between strain and motion, or from potential to kinetic energy. This efficiency is limited for a given material by the viscous dissipation of energy in the elastic straining process, and is correspondingly limited in the case of oscillatory electric circuits by the loss of energy in electrical resistance. One form of definition is that Q is the ratio of energy stored to energy dissipated, per cycle.

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Among elastic substances, quartz has, to the greatest degree known, this property of enabling high Q to be attained. This is equivalent to saying that a bell made of quartz, mounted in such a way that in "sounding" it does not transmit vibrations to its supports, and contained in an evacuated housing so that it radiates no sound, would ring longer, once struck, than if made of any other material. The values of Q measured for cylindrical rings of quartz vibrating at supersonic frequencies have run as high as several million, indicating specifically that the quartz ring, once started in its resonant vibrations, but without being further driven, completes several million vibrations during the time of the decay of its vibration to a value about one-third of the original amplitude (to a fraction equal to the reciprocal of the base of the natural logarithms instead of $\frac{1}{3}$, to be exact).

Not only do quartz resonators have the highest Q's known but they exceed those for the best of other measured elastic substances a hundredfold. Furthermore, the Q's of the best resonant electrical wire-circuits are less than a hundredth of those attainable in quartz resonators. Quartz crystals used in communication equipment commonly have Q's in the hundred thousands, the values depending upon the mountings, cleanliness, and precision of manufacture.

It is, in the last analysis, the degree of long-ringing power which determines the ability of any type of element to attain precision in controlling the frequency of an oscillating device. Looked at another way, Q is the measure of frequency-selectivity; that is, of the ability of a device to discriminate between frequencies nearly alike. A quartz element can be made a hundred times more sharply frequency-selective than elements of any other known material and is thus to the same degree more effective than any other device in holding the frequency of an oscillatory system, such as a radio oscillator, to the frequency preferred by the controlling element, in this case the quartz. The property of quartz under discussion is sometimes stated in terms of inverse properties such as the viscosity of the material and the damping of the vibration. Quartz has very low values for these.

B. *The Piezoelectric Property*

It is axiomatic that only materials which have electric properties can serve as circuit elements. Glass and mica are used as the dielectrics of electrical condensers owing to their characteristic electrical response to electric fields. Except for negligibly small mechanical deformations, known under the term electrostriction, the strains which simple dielectrics experience in an electric field are purely electrical. Quartz, also, has dielectric properties. But in addition, quartz in its crystalline form is piezoelectric, and experiences a second and more significant mechanical deformation under the influence of an electric field. This mechanical strain at any point in the quartz may be extensional (or compressional) and/or of a shear type depending upon the direction of the applied electric field in relation to the crystallographic axes.

The piezoelectric effect is a reversible one so that conversely when crystalline quartz is strained mechanically, there is called into being within the quartz an electrical polarization of the material which tends to set up charges on certain surfaces of the crystal or upon any nearby electrodes. It is its piezoelectric property which underlies the use of crystalline quartz in resonant circuit elements and is responsible for its affecting the circuit in another way than through its dielectric effect.

A piezoelectric element is thus more than a simple electrical condenser. The basic circuit effect is that in addition to currents through the quartz which are characteristic of its behavior as a dielectric, currents also flow through it as it deforms. The magnitude of this additional current is greater as the rate of deforming is more rapid, and the current reverses in direction as the rate of strain reverses. Thus, when any piezoelectric circuit element is in vibration, alternating currents flow through the quartz and through associated circuit elements as if there were an alternating electric generator hidden within the quartz and running in step with the changing of strain of the vibration, a generator which produces circuit reactions quite analogous to the more familiar counter e.m.f.'s of self-induction and other circuit properties. Although picturing the vibrating piece of quartz as an electric generator is particularly convenient when the energy which drives the vibration comes from outside of the circuit, as from sound waves impinging upon the quartz, its portrayal as a purely reactive circuit element such as a resonator is more appropriate for its radio uses.

C. *The Piezoelectric Resonator*

The flow of electric currents in circuits depends wholly upon voltages and such circuit elements as resistance, capacitance, and inductance. Following out of the fact that the deforming of a quartz crystal is directly related to the flow of current in its part of the circuit, the effect on the current in the circuit is as if the quartz element were some fixed combination of conventional circuit elements. The magnitudes in the combination can be expressed in conventional measures, but they represent values which are so far out of the ordinary that the equivalent circuit elements which would duplicate the circuit reaction are far beyond the ability of electrical engineers to construct physically. Piezoelectric currents of quartz elements are inherently small but are multiplied by the phenomenon of resonance. Yet even at resonance they are limited by the fracture strain of the material.

In one sense, the most significant fact regarding the equivalent circuit elements for a quartz mechanical resonator is that they constitute a tuned circuit, in this case an electrical resonator, and that this tuned circuit has a value for Q, to indicate its electrical frequency-selectivity, which is the same as that which characterizes the mechanical vibrating device. As stated, this value may be a hundred or more times as large as for the best wire-made electric circuits that could be built.

It is in the nature of the interaction of resonator and circuit that the more loosely the resonator may be coupled to the circuit and still affect it, the more rigidly is the resonator's own uniqueness maintained as a frequency of reference unaffected by the circuit. Resonator reaction on the circuit may remain effective through a more extreme loosening of the coupling the higher the Q of the resonator.

When quartz is used as a resonator in a radio circuit, no direct use is made of the mechanical vibration which is driven piezoelectrically by the circuit, or of the fact that the storage of the energy of vibration is accomplished mechanically rather than electrically, but only of the electrical reaction of the mechanical vibration back on the driving circuit, again through the piezoelectric effect. In other applications, piezoelectric elements serve to couple an electrical and a mechanical medium. Thus, in a loud-speaker or in a supersonic generator, piezoelectric crystals drive sound waves in an acoustic medium through the mechanical strains and motions which are set up in the piezoelectric material by an applied electric field. Conversely, piezoelectric crystals serve as microphones, in which sound waves in the fluid medium are themselves responsible for producing mechanical strains in the crystal, which in turn are inherently accompanied by voltages set up piezoelectrically. These latter drive currents in the rest of the circuit of which the piezoelectric element is a part. In these applications, the crystal's function is as a transducer, so-called in communication engineering literature, to lead vibrational energy from one medium to the other, as from the electric circuit to the acoustic medium, or vice versa. In contrast to the resonator application, which is of principal concern in radio and telephone communication, and in control of frequency, it is not usually important that the crystal element which is used as a transducer have a high Q. One crystal used in transducer applications is Rochelle salt, which has elastic properties very inferior to those of quartz. It has, however, much larger piezoelectric properties; larger strain results from an equal field, and greater current through the crystal accompanies an equal rate of strain.

D. *Other Factors in the Common Circuit Use of Quartz*

The mechanical resonance frequencies in a piece of quartz depend upon its size. As in other systems of stationary waves, the period of the vibration is the time required for the mechanical wave in the material to complete the round-trip between opposing boundaries of the system. The speed of elastic-wave travel in quartz is such that round-trips in pieces whose frequency-determining dimensions lie in the range between a fraction of a millimeter and a few inches, require lengths of time which correspond to the intervals between successive alternations of currents of the frequencies used in radio. Thus, fortunately, quartz resonators which are tuned to radio frequencies have convenient dimensions. Small crystals are associated with high frequencies and the whole gamut of frequencies for which quartz is used runs from a couple of kilocycles per second (kc/sec), which is a voice frequency rather than a radio frequency, to a hundred or more megacycles per second (mc/sec). The shorter the dimension of a crystal along which the standing elastic wave is set up, the higher is the radio frequency to which the crystal is resonant, although the scheme is sometimes adopted of getting resonance at higher frequencies by letting the standing wave system be a number of half waves long instead of one.

No small factor in the importance of quartz crystals as standards of frequency in radio communication is the excellence of its other properties in addition to the elasticity already referred to; notably, its chemical stability and permanence under a wide range of conditions, its low solubility, and its mechanical hardness. Again, some of its elastic compliances, have positive temperature coefficients while others have negative, and this offers great possibilities for balancing compensating temperature effects on frequency against one another. Highly important to its recent extensive military application has been the fact that quartz was available, and the further fact that there has existed wide knowledge of its detailed properties following years of intensive investigation of its resonator behavior.

II. THE MECHANISM OF CIRCUIT INFLUENCE BY QUARTZ

A. *The Piezoelectric Strain-Current Relation*

It has been pointed out that electric current flows through the quartz during the changing of its state of strain, that the quartz unit may be regarded as a circuit element, and that as such it has both dielectric and resonator properties. If it were possible to so firmly clamp the piece of quartz that the circuit voltages could produce no mechanical strains, the crystal would serve merely as an ordinary condenser. The piezoelectric currents are over and above those due to this condenser element of clamped quartz, and are properly regarded as motional currents from their association with the motion of the quartz. Analogous motional currents and motional impedance properties are familiar in the treatment of loudspeakers.

In its electric circuit representation, the effect of freedom of vibrational motion of the quartz shows up as a second branch of the circuit, bypassing the condenser. This parallel branch is fictional rather than physical, but the laws governing the flow of current in the rest of the circuit include the reactive effects of such an equivalent circuit having a characteristic motional impedance. The piezoelectric property of the quartz may be thought of as serving a sort of mirror-like function, whereby the current flowing in the circuit leading up to the quartz sees, as opposition or impedance to its flow, an electrical image of the mechanical opposition which the quartz offers to the forces which the applied voltage has set up to drive it. Stated again, the piezoelectric effect offers a translating formula whereby mechanical properties develop electrical properties in the circuit in proportion to their own magnitudes. It has long been a custom to think of alternating current circuit behavior in terms of the vibration of mechanical analogies. In this case, however, we have more than an analogous behavior; the mass, stiffness and viscosity of the quartz actually appear as parameters in the electric circuit multiplied by appropriate coupling factors; similarly, these coupling factors interrelate electromotive force and mechanomotive force, electric current and strain velocity. The quantitative basis of these associated effects is in each case a piezoelectric constant of the quartz modified by specific dimensions, by the type of strain involved in the vibration, and by the direction of the electric field in the quartz to which the external circuit is made susceptible.

The magnitude of the current in this parallel motional branch, in other words the piezoelectric current, is usually negligibly small compared to the dielectric current of the condenser except when the frequency of the driving voltage is close to a resonance response frequency of the piece of quartz. At such a frequency the extraordinarily large value of the Q multiplies or enhances the current in the imaginary piezoelectric branch by a factor of the same order of magnitude as the Q. That is, the effect of high Q is to reduce greatly the magnitude of the electrical impedance of the piezoelectric branch at resonance. With Q's of the order of 100,000, the piezoelectric responses, both the current and the mechanical strains, usually become large enough at resonance to overshadow completely the dielectric effect. It is the mechanical resonance effect alone, this multiplication by large Q, which brings the otherwise exceedingly feeble piezoelectric currents into usefulness in the radio field.

B. *The Several Circuit Uses of a Crystal Resonator*

Quartz crystals are sometimes confusingly spoken of as being either oscillators, calibrators, resonators, or filter crystals. They are all resonators; but some are used in oscillator-circuits to play a part in controlling their frequencies, while others are used to show their resonance effects while the driving voltage is controlled in frequency by some source independent of the crystal.

In an oscillator where the natural ability of the quartz resonator to establish the timing of pulses makes it successful in imposing its mechanical characteristics on the less headstrong remainder of the circuit, the response of the crystal, its current in relation to voltage and frequency, is no different at any given frequency from that which it would demonstrate if the control of the frequency were in other hands entirely. The piezoelectric circuit response of the crystal to whatever voltages are placed upon it is always purely that of a resonator. Its reaction is characteristic of all resonator reaction whether electrical, mechanical, or acoustical, and includes an amplitude and phase-angle of response which depend on the relative frequencies of resonance and of drive in relation to the Q of the resonator.

In another common use, piezoelectric crystals are elements of the electric wave-filters which serve in carrier telephony, and here again the function of the quartz is merely to respond as a resonator. The crystal, in this case, is no more the filter than is the crystal in the oscillator-circuit the oscillator. The confusion of terms presumably comes about from the synonymous terms "oscillation" and "vibration." Both circuit and crystal are in oscillation. Best usage, however, reserves the term "oscillator" to mean oscillation generator. A complete vacuum tube circuit, with or without a crystal element, serves as the oscillator or generator. Although the crystal "oscillates," "vibrates" is preferred, for the crystal is a resonator.

A principal difference between a vacuum tube oscillator which has a crystal in a place of control of frequency and one not provided with crystal-control lies in the range of frequencies over which the oscillation may be varied by adjustment of some variable element such as the tuning condenser or by chance circuit disturbances. As the condenser-dial is turned, the oscillation frequency of a circuit not controlled by a crystal may usually be varied over a wide range, commonly severalfold.

C. *Crystal-Controlled Oscillators*

There are many types and designs of oscillator circuits for using the crystal's controlling influence. The more common types, at least among those used in radio communication, place the crystal unit across a condenser in the circuit which, thus being in parallel with the crystal dielectric, serves merely to increase the total capacitance shunting the motional properties of the crystal. This combination has an extreme variation of its impedance over a very small frequency range, for within a region a hundredth of a per cent or so above the frequency of mechanical resonance of the piece of quartz (imagined isolated) the impedance of this parallel combination may vary tens of thousandfold. It is the extreme sensitivity to frequency which the impedance (strictly the reactance component) of the crystal shows, whether shunted or not, which is responsible for the extreme variation of its reaction on any oscillator which is not too remotely coupled to it and whose frequency would be likely to vary through the resonance region of the crystal if uncontrolled. Upon this feature depend its control both of oscillator frequency and of the sharpness of boundaries of the bands of electrical filters.

It is the nature of one type of oscillator-circuit that it oscillates at the frequency at which this parallel combination of motional and dielectric branches, including the capacitance external to the crystal, reaches its highest impedance. Another type of circuit oscillates at the frequency of lowest impedance of the crystal, which is its frequency of mechanical resonance. Because of the character of the resonance effects in the equivalent circuit, these two types of oscillators are commonly described as using the crystal in parallel resonance and in series resonance, respectively.

A principal example of the series resonance type of oscillator is the bridge-type designed by Meachen which is used principally for oscillations of highest precision of frequency. In another, the crystal is placed in series with the coil of the tuned circuit of a Colpitts oscillator.

The parallel type of oscillator appears in two common forms. In one, the crystal is located between the grid and the filament of the vacuum tube; in the other, between grid and plate. The frequency of parallel resonance is always higher than that of series resonance of the crystal;

thus the same crystal would control oscillations of slightly different frequencies in the series- and in the parallel-resonant connections. The amount by which the parallel-resonant frequency exceeds the series-resonant frequency for a given crystal depends upon the dielectric capacitance of the quartz and that of the external circuit, and may be as large as several kilocycles per second for an eight megacycle per second crystal, or may be as small as, say, fifty cycles per second. The larger the total parallel capacitance, the smaller is this difference in frequency between series and parallel resonances.

Differences in equivalent motional-impedance properties exist among crystals of approximately equal size and shape because of differences in their effective piezoelectric properties, such as arise from twinning, or from differences in cut or in the details of the complex vibration pattern. As a result, the displacement of the parallel-resonant oscillation frequency from series resonance for the same total capacitance load may vary from crystal to crystal of the same type. It is for this reason that a group of crystals all ground to give identical oscillation frequencies in a given oscillator of the parallel resonance type may show a considerable spread in their oscillation frequencies in other oscillator-circuits having a different capacitance, whether by design, or resulting from manufacturing tolerances. For such a group of crystals, their mechanical resonances might show a considerably larger scattering. Unless special attention in manufacture is given to the magnitude of the difference in frequency to be experienced in oscillation in a series-resonant connection and in a parallel connection across some stated capacitance, it cannot be assumed that close agreement between two crystals in controlling frequency in one circuit will result also in their agreement in frequency in a different circuit. It will perhaps emphasize the point to state that it is the accepted practice in marking the frequency on crystal units for use in communication equipment to indicate the nominal frequency of oscillation in a standard oscillating circuit which places a stated capacitance or a stated characteristic impedance load across the crystal. This nominal frequency is not a characteristic of the crystal's mechanical resonance alone, but assumes its use in a specific electrical connection, and the crystal's use is valid only in connection with an equivalent load.

It may be noted in further reference to the valid use of a crystal that there are slight differences in the frequency position of the crystal's resonance itself which are associated with the amplitude of vibration of the quartz, but whose explanation is not as yet clear. Over and above those changes which can be ascribed to the heating of the quartz through its own dissipation of vibrational energy or that in the mounting mechanism, there appears to be a lowering of the resonant frequency as well as a corresponding change in the impedance properties of the crystal at any fixed frequency near resonance when the crystal is introduced into a second circuit which is identical to the first except that it places a greater voltage across the crystal. The change in frequency is not large enough to be troublesome when frequency tolerances are large, but will become a factor in the application of crystals of tolerances greatly reduced in comparison with those which are now common in communication equipment. It can be a critical factor in the adjustment and frequency stabilities of precision crystals and imposes stringent factors in the using circuit, requiring that crystal currents be kept very small.

III. The Importance of Constant Frequencies in Communication

A. *Carrier Current Versus Simple Telephone Circuits*

About thirty years ago modern telephony began to multiply significantly the number of conversations which could make simultaneous use of a pair of telephone wires. By the introduction of several carrier currents, each of a frequency well above the highest frequencies of the voice, and each carrier frequency well separated from the other, it became possible, by impressing the characteristics of several voices upon these several carriers, to guide these latter by their frequencies rather than by the ordinary voice-current wires so as to offer channels of communication which transcended the single pair of telephone wires. With the voice impressed upon a high frequency carrier current, a branch circuit selective of the frequency range of that particular carrier would pass that voice into the branch and reject another which was impressed upon a different carrier frequency. In this manner, the pair of telephone wires reaching from city to city in long distance telephony frequently serves as the common circuit over which a number of channels of communication, which have been gathered in over separate pairs of wires from local subscribers, are routed between remote points, each channel having a different characteristic frequency range on the common part of the circuit.

The key to the successful operation of carrier telephony is the ability to translate the frequencies which are present in the voice currents to a corresponding band of frequencies placed higher in the frequency spectrum, that is, to the vicinity of the carrier frequency. And again, at the receiving end of the circuit, the voice on the carrier must be stepped down again in frequency to the voice range. The process known in communication engineering as modulation takes care of the lifting up of a band of voice frequencies to the level of the carrier frequency or of dropping it down again to voice frequency levels.

B. *The Carrier Principle in Radio Telephony*

Wireless communication is based upon the property of electric circuits to radiate their energy more effectively as the frequency of the electric current is higher. Low frequency currents have relatively little effect away from the immediate vicinity of the wires of their circuits. For each high frequency a fraction of the power of the current, which depends on the geometry of the circuit as well as the frequency, departs from the vicinity of the circuit and radiates off as radio waves. The voice is transmitted in radio as a modulation superimposed on the high-frequency oscillation of the radio transmitter and radiates from the station impressed upon this carrier radio wave. The frequencies to which voice frequencies are stepped-up in radio vary from a few tens of thousands to hundreds of millions of cycles per second. Successful operation imposes close restrictions or control of frequencies in the step-up and step-down processes.

C. *Precision of Frequencies Essential*

Enough has been stated to suggest that there is need for controlling the frequencies of radio oscillations which are to be used as carriers to some degree of precision. The susceptibility of ordinary circuit type oscillators to disturbing influences, which cause frequencies to change or to wander, is relieved by putting the control in the hands of a crystal, and thus packing into a very small space the assurance that frequency drift will be within narrower limits.

In helping to set the path which the message on a given carrier will take, crystals sometimes play more than one part. They not only determine the frequency of the carrier and hold it so that it does not shift to the point of possible confusion of identity with and interference with an adjacent carrier (nearby frequency), but they also sharpen up the selectivity of filters so as to let through to a given branch circuit only the intended modulated carrier while discriminating against others. The crystal makes possible in both of these ways the closer packing of channels among the available frequencies of the radio spectrum. Thus not only does one group of crystals determine the frequency gate-posts between which the modulated carrier must pass, but another crystal establishes the carrier frequency which assures that when modulated it will successfully pass through the gate.

In some uses it is the vibratory current in the crystal which after being built up to greater power in later stages of the transmitter becomes the actual radiation. In others, the crystal in a feeble oscillator-circuit serves merely to establish a reference frequency against which the transmitter oscillation is compared, or to which the latter is either manually or automatically adjusted. Crystals for use in such reference oscillators are often referred to as calibrator crystals. Their frequencies may be used to calibrate reference points on the dial of the master variable oscillator so that a chart of its frequency, in terms of dial settings, can be used with greater precision because of the corrections determined for these reference points.

Another use of a crystal oscillator is to keep time rather than to furnish a frequency for reference, although the two are alike in their aim for constancy of frequency. When its successive oscillations are to be added up and a count kept, as in the crystal clock, the interest may be in the total count of vibrations rather than in the instantaneous rate of vibrations. The GT-cut crystal in a bridge-type oscillator has proven itself to be the most precise standard clock. An inherent feature of the crystal clock, in view of the small sizes of pieces of quartz available, is the existence in the clock oscillation circuit of small subdivisions of seconds, commonly as small as a hundred-thousandth of a second. These are valuable as reference points in time, whereas it would be impracticable to identify any phase of the swing of a seconds pendulum to any such precise instant.

D. *Tactical Benefits from Crystal Control*

Where two radio sets must communicate with one another, it is a relatively simple matter, if somewhat time consuming, for either operator to vary his tuning in order to locate the frequency which the other is using and thus to establish communication. The resulting frequency is, however, likely not to be in any absolute sense that which has been assigned for the channel of their communication unless some reliable reference frequency is available to them in determining the channel. The process of searching for the signal from the other party offers, furthermore, the surest means of upsetting an orderly plan of channel spacing and of introducing, as a consequence, interference between stations. In the absence of good frequency control, considerable drifting or swinging of carrier frequencies around their nominal value is the rule and such chance drifting of one carrier only makes more necessary the search, or adjustment, on the part of the second station. The function of the crystal in controlling the frequency of the radio set is to assure that the radiation sent out by the first station is on the allotted channel, and also, when so used, to assure that the receiving station is attuned to that same frequency without search. The receiving crystal is of such a frequency as to step down just the particular frequencies of the carrier channel it is intended to receive to the level of an established lower frequency gate or filter so that after its isolation from possibly confusing frequencies its conversion into voice may proceed.

When a number of radio sets must be brought into frequency agreement so that each can talk with any other of the group, the process of successive adjustment of all to the chance or intentional changes that may occur in one of the sets becomes extremely laborious and time consuming. It is here, particularly, that the advantages of fixed frequency location of channels by crystals are predominant. By using crystals, positive communication is provided without search on the instant of bringing the appropriate crystals into the several circuits. Furthermore, the energy radiated by each of the sets is within the allotted channel and any interference which these waves cause to other communications is characteristic of the system of channel allotment rather than of an uncontrolled swing of the carriers.

The speed and simplicity of the operation of crystal-controlled circuits, the fact that they require little training in their use and usually do not require operators experienced in the technical side of radio, and their ready adaptability to push-button operation and selection of channels are large factors in the popularity of crystal-controlled equipment. Some simplification of circuit design is made possible by making available to each set a number of crystals so as to give it access to a multiplicity of communication channels, if not to the infinite degrees of off-channel operation.

There may be, however, circumstances which warrant the shift in channel frequency to a nearby free region when the assigned operating frequency suffers from interference. The designer must usually make his choice between providing for communication on any frequency in the range without crystals and providing only predetermined channels. Only for the latter case may crystals be provided, since crystals are not available with sufficiently variable frequency for the former.

IV. THE IMPORTANCE OF THE ORIENTATION OF QUARTZ RADIO ELEMENTS

A. *Orientation for Desired Mechanical Properties*

The dependence of Young's modulus and other elastic constants of quartz upon the direction of the stress in relation to the crystallographic axes is well-known. The detailed mechanism of the mechanical vibration of a piece of quartz in any mode involves the values of the elastic constants which relate to that mode, the density of the quartz, and the geometry and dimensions of the piece, as well as the constraints imposed by the mounting. The dependence of a particular elastic constant upon direction results in the variation of the resonance frequency which it determines, with slight change in angles of cut. Owing to the interlocking dependence of the main strain upon accompanying minor strains in other directions the complete detail of the combined motion and the frequency of the resonance actually depend simultaneously, in most cases, upon several elastic constants and dimensions.

A mechanical mode of vibration is, except in what are usually wholly negligible ways, entirely a mechanical problem, and the ability of a plate or bar to show mechanical resonance at a given frequency depends upon the appropriateness of elastic constants, orientation, and dimensions to the setting up of a standing wave system at that frequency. The manner of applying the external forces which might set up the proper strains is not important to the description of a mode of possible resonance, although before such a mode can be physically excited some driving mechanism must be found.

It is characteristic of elastic solids that under a stress applied in one direction, the strain which that stress produces in this same direction is accompanied by other strains in other directions. The most familiar example of such strains is the sidewise shrinking of a bar or wire when stretched lengthwise, a property expressed in Poisson's ratio for the material. In crystalline media, as distinguished from isotropic, these cross-strains may appear unsymmetrically as is evident from a study of the elastic constants of the crystal. They become important in quartz crystal vibration where in resonance the cross-strain may become large enough to be a significant fraction of the main-strain. If the cross dimensions are appropriate to their establishing a second set of standing waves superimposed upon the first, the interlocking effects between these two become exceedingly complex. Dust patterns and other means of detailed study of the motion over the face of a vibrating quartz plate indicate that the modes of motion are very complicated indeed, and the familiar dust patterns on Chladni plates are intricate enough without the additional effects of aeolotropism. Because of the minute scale of the detail of the wave patterns on a quartz plate, they shift with the slightest change in crystallographic orientation or in size or shape, and the circuit behavior of the crystal changes accordingly.

B. *Predimensioning: Precise Duplication of Orientation and Dimensions*

The precise mathematical solution of such a complex vibrational system is probably hopeless. The practical solution for radio quartz plates is to prepare a model plate, adjusting it by methods which are partially "cut and try" until it gives satisfactory performance in the radio circuit, or better, gives the combination of intrinsic properties desired. This model is then copied in production, exactly duplicating the orientation, geometric shape, and the dimensions. This use of a model and precisely copying it is commonly referred to as the predimensioning method of manufacturing quartz plates as distinct from the custom-making adjustment of each crystal separately until it gives adequate performance in the radio circuit. The former method results in a degree of homogeneity between crystals which are produced to the same dimensions which is entirely lacking in the latter method of production.

C. *The Effects of Orientation on Piezoelectric Properties*

Although, as has been indicated, the mechanical vibrations of a quartz plate are characteristic of mechanical properties alone, the necessity of coupling such mechanical vibrations to an electric circuit requires the use of preferred directions in the quartz for the principal strains in the vibration which can produce piezoelectric currents at suitable electrodes. The optic-axis direction, for example, is entirely devoid of electrical coupling to strains in the quartz since this direction shows no piezoelectric properties. Vibration of a plate cut perpendicular to the *c*- or Z-axis, a Z-cut, as it would be called, can neither be driven electrically by a field along Z, nor can electrical effects of any such vibration be picked up by electrodes symmetrically placed on Z-faces of the plate.

In general, electric fields which are perpendicular to the optic axis are the most strongly coupled to mechanical strains, although even then only to certain particular strains. Quartz plates cut to take advantage of these maximum directions of the piezoelectric effect are the X-cut and Y-cut plates, named from the fact that the normals to their principal surfaces are parallel to the X- and Y-axes of the quartz, respectively, the *a*- axis of the crystallographer and its normal. It is in the nature of the piezoelectric properties of quartz that electric fields parallel to an X-axis, as in an X-cut plate, are associated with stresses or strains of a compressional type along the directions of X and Y, one being extensional while the other is compressional, as well as with an added shear type of strain. The shearing layers sliding with respect to each other may be thought of as either XY-planes showing relative displacements in the Y-direction, or as XZ-planes showing relative displacements along Z; in any case the displacements in the shear are perpendicular to X.

The effect of a Y-field, on the other hand, as in a Y-cut plate, is to produce two superimposed shearing stresses, one in which all displacements are perpendicular to Y, and the other all perpendicular to Z. The one may be thought of as the sliding of neighboring YZ-planes in the Z-direction, or alternatively of the sliding of XY-planes in the X-direction. The other shear may be viewed as the relative displacement of XZ-planes in the X-direction or YZ-planes in the Y-direction.

The several stresses and strains are related through the so-called piezoelectric moduli and/or piezoelectric constants to the electric polarization of the quartz, some to the X- and some to the Y-component of the polarization, and thus to the corresponding electric fields or surface charges. Along with the orientation of a vibrating quartz plate and the detailed character of the mode of the vibration, these constants determine the magnitudes of piezoelectric currents within the quartz, between the electrodes, in relation to the changing mechanical strains in the plate.

D. *The Oblique Cuts*

The continuing development of the art of crystal control has led to the use of quartz plates no longer normal to the plane of the X- and Y-axes. In general, the piezoelectric coupling between vibration and circuit is less as the orientation of these plates departs farther from the normal, although the mechanical properties show no corresponding deterioration, but only change. Specific orientations are selected to take advantage of the compensating effects between the temperature coefficients of the several elastic properties which play parts in the vibration so that the resonance frequency of a plate may remain much more constant over a range of temperatures than does any one elastic constant of quartz. At certain other inclinations, the cross-coupling of one type of elastic strain to another vanishes, and plates so cut as to take advantage of this are much more free from the problems of complexity of the mode of vibration than plates not so cut and may even retain this freedom over a wide temperature range.

Shear vibration is the common type among those oblique cuts which may be thought of as derived from Y-cut plates by rotation about X. Longitudinal modes characterize those bars which are derived by a rotation, about X and about their length, from X-cut bars whose length lies along Y. The several orientations of these inclined "cuts" need, in general, to be held with great precision in order to take full advantage of the temperature compensating mechanism or of the interrelations between modes, although some small degree of misorientation can often be balanced by slight modification of the dimensions.

V. The Ranges of the Characteristics in Quartz Crystal Units

In a survey paper, intended for readers in fields other than physics or radio, it seems desirable to include a brief analysis of each of the many variables which one finds among crystal units, in order to permit the reader to classify more readily the many types of crystal units which he may encounter. Except when the holder is made of clear glass, as is sometimes the case, little is evident as to the crystal itself from the outside of the crystal holder, of course, except perhaps for such markings as indicate the nominal frequency of the oscillation which the crystal may be intended to control. The limitations of space to which the unit may be adapted and the characteristics of the mechanical fitting to the radio set will be evident, but the size of the holder may be misleading as to the size of the piece of quartz which it contains, and its external features give no indication of the type of mounting.

A. *Size of Crystal Plate or Bar*

Quartz plates range in thickness from six or eight thousandths to half an inch, with fifteen to thirty thousandths as the sizes which have probably been made in the greatest numbers. Face shapes are most commonly square, or rectangular near square, with dimensions of the order of half an inch on a side, while plates as small as one eighth inch square and as large as two inches square are standard items. Bars are used at present in much smaller numbers than plates, although this is probably owing to the frequency range, three to nine megacycles per second, which has seen greatest recent application. Filter uses are primarily for bars. Bar sizes have run from about $3/8'' \times 1/8'' \times 1/16''$ to $3'' \times 1/2'' \times 1/8''$, with the most common sizes in the middle of the range.

B. *Frequency*

Crystals are made to resonate at frequencies scattered all the way from a few thousand to more than one hundred million vibrations per second. All crystals show more than one frequency of resonant response; most have a large number. The predominance of the desired resonance in its particular region of the spectrum must usually be supplemented by circuit preferences for that region over others in order that the crystal-controlled oscillator shall make use of the intended resonance. This resonance may not be a fundamental mode of its type, in which, of course, the frequency determining dimension is a half wavelength in the quartz, but may be some overtone in which the crystal is several half wavelengths thick, or long, as the case may be. In overtone modes the crystal acts as if subdivided into sections, each of which vibrates almost identically, with alternate sections in opposite phase.

C. *Type of Vibration*

In some crystals the vibration is predominantly extensional and contractional along a given dimension of the plate or bar. In others, the motion is of shear type. In these latter the shear may be, in some cases, a distortion of the major faces of the plate, while in other cases the undistorted major faces may be thought of as sliding with respect to each other along their planes. These types of shear are referred to as face-shear and thickness-shear, respectively.

Still other crystals are used for their resonance in flexural vibration. Here again there are two planes in which the flexure may occur, as well as long series of overtone modes for each. A crystal cut like a thin lath may bend the easy way and resonate at a very low fundamental frequency, or it may select the stiffer flexure in a plane at right angles to this and a fundamental mode of resonance at a much higher frequency. The type of vibration which is excited in a given case depends partly upon the method of mounting and the points of constraint which this determines, and also upon the cut and the disposition of electrodes. More elaborate arrays of these latter are used for exciting flexural modes, and likewise for overtones of most types, than for the fundamental modes of shear or of extension.

If the electric field is applied in the direction of the dimension which determines the length of the principal standing wave system, only odd overtones can, in general, be excited, for in only these cases are opposite charges on the two electrodes produced by the motion. A well-known crystal of this type is that frequently called the "third-harmonic" AT-cut plate. This vibrates in thickness-shear in three layers at a frequency which may be 12 mc/sec, whereas the plate is of a thickness to vibrate at about 4 mc/sec if its fundamental thickness-shear resonance is excited. Frequencies of oscillation circuits as high as 197 mc/sec have been controlled using the fifteenth thickness mode. Special types of oscillator-circuits are required to select the desired overtone mode of vibration from among the extensive series of possible modes.

The practice of designating these vibrations as "harmonics" may lead to some confusion between mechanical overtones of crystals and the harmonics of the vacuum tube oscillator. As is the case with all vibrating solids, the several overtones of a given fundamental type of vibration do not follow the harmonic series strictly. On the other hand, there are cases in which crystal oscillator-circuits are designed to emphasize some exact harmonic of the frequency of the crystal oscillation, as where the harmonic is produced in the process of distortion of the alternating current by the vacuum tube. In the one case, the crystal and circuit oscillate at the overtone frequency and neither the motion nor the current at the fundamental frequency is involved. In the other, the crystal and circuit oscillate at some crystal frequency and a branch circuit, or perhaps some later stage of amplification, selects a harmonic which has developed in tube distortion. The designation "third mode (of thickness-shear)," for the case described in the preceding paragraph, would avoid the confusion and reserve the term "harmonic" for the case of distortion of alternating current wave-form, where its use is well-established and is proper.

The following breakdown of the frequency spectrum into the modes of vibration which are perhaps most commonly used in the several frequency ranges may be found useful:

Frequency range (cycles/sec)	*Type of Vibration*
2,000– 30,000	Flexure
30,000– 100,000	Extension and compression of bars
100,000– 1,000,000	Face-shears of plates
1,000,000– 15,000,000	Fundamental thickness-shears of plates
15,000,000–100,000,000	Higher-order modes of thickness-shear

There are no sharp boundaries between these ranges, and a good deal of overlapping exists. The figures given are intended to be merely suggestive.

In the complex vibration which characterizes many of the common and useful resonances, particularly the thickness modes of plates, there is superimposed upon the predominant type of motion a complex superstructure of overtones of other fundamental modes, some of which represent other types of vibration, and many of which are resonances along a different dimension than that of the principal motion. The entire plate vibrates at one frequency but the motion, in some parts, may be either just opposite to that in other parts, or perhaps greatly increased or greatly reduced in comparison with neighboring regions.

D. *"Cuts"*

The orientation of a quartz plate or bar is referred to either by the angles which the normal to a major face makes with the crystallographic axes or by a characteristic name which indicates a cut having special properties. Thus the AT- and BT-cut plates are inclined +54° 45′ and −41°, respectively, from a Z-cut, the rotation being about X. These cuts determine frequencies for their fundamental thickness-shear modes which have relatively small dependence upon temperature. Similarly, CT- and DT-cut plates are inclined from a Z-cut by angles of +52° and −38°, respectively, about X, and show small dependence of their face-shear frequencies upon temperature. The convention of signs now used gives positive signs to counter-clockwise rotation as seen from the positive end of the axis of rotation for a right-handed axial system, clockwise for a left. A right-handed axial system is used for right quartz, and left-handed for left quartz. The positive end of the X-axis in either quartz is identified by an edge of the prism at the ends of which *s*- and *x*-faces are possible.

The angles given in this paper are not those by which the "cuts" referred to are best known, but rather their complements. The more commonly used designations, such as +35° 15′ for AT- and −49° for BT-cuts, are the inclinations of the plates themselves to the Z-axis rather than of their normals, as are used in this paper.

Considerable confusion regarding the definitions of right and left quartz and the senses of axes and of positive rotation pervaded the radio engineering literature on quartz crystals prior to 1940, when general agreement was reached to use the mineralogists' definitions regarding the nomenclature of the enantiomorphic forms and the convention of signs described above. An Institute of Radio Engineers Committee Report regarding such a standard convention is in process of publication. In order to provide an unambiguous system of specification of the orientation for quartz plates or bars cut at any inclination, three rotations are listed about three axes, to be performed in order. By these, starting from the standard reference position of a Z-cut rectangular plate with its length along X, any position of the plate may be specified. All rotations of the plate, assumed to be a rectangular parallelopiped, carry with them a rotated set of axes parallel to the edges of the plate. In their rotated position these axes are primed, as X′, Y′, and Z′ after a first, and double primed after a second rotation. As already stated, for a right quartz, and a right-axial system, positive angles appear as counter-clockwise when viewed from the positive end of axis of rotation.

The first rotation is performed about Z, and is called ϕ; the second, θ, about Y′; and the third, ψ, about Z″, which is also Z‴. Although a final designation of orientation is unambiguous, it is possible to arrive at two or more designations for the same physical orientation depending upon the choice of a plus or minus 90° angle for ϕ in the first rotation by which Y′ is placed along X preparatory to the second rotation. Thus the BT-cut plate may be alternatively designated +90°, +41°, 0° (or 90°) or −90°, −41°, 0° (or 90°). There is some reason for preferring the second which brings Y′ into the same sense as X instead of the first where Y′ is in opposite sense to X. The −41° designation of θ is also in agreement with the numerical description for the BT-cut given earlier where only the conventional mathematical procedures for a right-handed axial system were used, without reference to the complete I.R.E. system of three rotations.

The use of a complete mirror image of the axial system and senses of angles to conform to the change in enantiomorphic form to left quartz provides that the BT-cut in a left quartz carries the identical set of angle indices, and each other cut, likewise, is identically described in either quartz.

Other well recognized cuts beyond those already described include GT-, ET-, FT-, and NT-cuts, and the so-called −18° and +5° bars, but reference should be made to other papers for characterization of their properties.

E. *Temperature Coefficients of Frequency*

The frequencies of crystal resonators vary with temperature at different rates at different temperatures, particularly where approximate constancy of frequency over an extended temperature range is secured by compensations within the quartz over that range, as in specific cuts. As a result, except where the crystal is to be held to the close vicinity of one temperature, the total variation of frequency is usually of more interest than the temperature coefficient at any one temperature. For curves showing the characteristic variation of frequency with temperature for the several cuts, the reader is referred to the original sources. The frequency of a BT-cut plate follows a curve which is roughly a parabola with the vertex showing the maximum frequency located at a mean temperature in the total temperature range of interest. The temperature at which this highest frequency occurs is controlled by choice of the precise orientation for the cut, being about 30°C. for a −41° BT-angle, and higher as this angle is larger. A departure of about 0.025% from the highest frequency is inherent in the characteristic curve for temperature variations of 70°C. either upward or down from the chosen mean temperature.

The frequency drift curve for varying temperatures which characterizes an AT-cut plate is less well determined. Its shape depends to a very considerable extent on the precise dimensions chosen unless the plate is very thin in comparison with its length and width. Judicious choice of dimensions appropriate to a specific orientation will permit a relatively flat frequency curve over a large part of the 145 Centigrade degree range for which many manufacturers are making crystals. The possibilities of considerably reduced frequency tolerances from using this cut in preference to the BT-cut for frequencies in the 5 to 10 mc/sec range have not yet been exploited. Units departing from a mean frequency less than twenty parts in a million over a temperature range 145 Centigrade degrees wide have been made, but manufacturing tolerances for this extreme frequency precision may well be too close to be met in production. The curve for any cut is susceptible to the modifying influences of cross-coupling for any arbitrary dimensions of the plate, especially when the plate is thick (a tenth or more of its length or width). This distortion is particularly common for AT-cut plates where the lower frequencies used often call for extreme thickness.

F. *Temperature Range*

In the military use of crystals, where frequently the crystal temperature is not controlled, the limits to which ambient temperatures may run force extreme requirements as to the limits of angular tolerances which can be permitted in manufacture. The characteristic curve for the BT-cut crystal is illustrative of the problem, for it makes no allowance for internal heating of the crystal caused by its vibration. Circuits which cause the crystal to dissipate more than a hundred milliwatts offer special difficulties on this score since the frequency follows the crystal temperature, of course, rather than the ambient for which frequency curves are commonly drawn. Also, the crystal unit may have to operate in an ambient of wider range than the radio set, as crystals are sometimes placed in compartments which warm up during the operation of the set. The extension of the crystal's operating range may become considerable if the set must operate from a cold start in the coldest weather as well as after a warm-up period of operation in the hottest weather. This requirement may well add 25 to 30 Centigrade degrees to the operating range, and the internal heating of an active crystal in a vigorously driven set an additional 25° or more. The BT-curve, although adequate for $\pm 0.02\%$ frequency limitations over 145 Centigrade degrees when proper design considerations are observed, scarcely permits the operating of crystals within these tolerances over the full range of $-55°$ to $+70°$C., which has been laid down as the ambient for some equipments themselves, unless care is taken to avoid extremely active crystals and to locate crystals within the equipment where they will not be warmed up by the tubes. It is the most active crystals which are driven most violently and, therefore, heat up the most. To include some allowance for compartment heating, crystals are often specified to maintain tolerances over the wider ambient temperature range of $-55°$ to $+90°$C.

G. *Load Circuit*

The circuits in connection with which crystal units are to operate impose very definite limitations on the properties of the crystal unit. One of the most important circuit characteristics is the impedance across which the crystal is to be connected. Many equipments which use the crystal in the parallel resonance connection place it across a capacitance in the circuit, and the magnitude of this capacitance determines the location on the crystal resonance curve of the frequency at which oscillation is to occur. Hence, in finishing crystals they are adjusted to provide the desired frequency when used in connection with the appropriate value of capacitance. Such values range from ten to a hundred or more micromicrofarads. It is characteristic of a fixed capacitance load that the magnitude of the load impedance varies inversely with the frequency. In multichannel equipments where a number of crystals are to be provided covering a wide range of frequencies, the lower frequency crystals are thus operating into a relatively large circuit reactance while the higher frequency crystals face a small reactance.

Some circuit designs, on the other hand, place an impedance across the crystal which follows a different law with frequency than that of a fixed capacitance, and the crystals made for such equipments require to be adjusted to frequency under the appropriate impedance load condition which is specified for the frequency in question. Among the several series of commonly used crystals in military communications, some are intended to operate across a stated capacitance for all crystal frequencies and others across a particular combination of inductance and capacitance. It is in cases such as the latter, particularly, that circuits which are exact copies of the oscillator in the using equipment need to be provided for the manufacturer of crystals in order that he may adjust each crystal to the circuit impedance which the "test set" shows at that frequency. It is somewhat more simple to duplicate from characteristics set down on paper the testing conditions where the circuit load is a fixed capacitance than for a more complex load.

H. *Activity*

In finishing crystals, the manufacturer is usually particularly concerned about producing units which are sufficiently "active" to meet the requirements of the using set. There has grown up out of this concern of the manufacturer, and also out of experience with "dead" crystals, a common belief that the most active crystal units are always the best, or that high activity itself will result in better performance of the radio equipment. Such is not the case. A crystal's activity depends upon its electrical impedance. In the design of circuits, all other elements than crystals have their impedances specified within both upper and lower tolerances, and a similar statement of the best value and of its upper and lower limits is equally desirable in the case of a crystal as a circuit element, as indeed is already done with precision crystals in the low frequency ranges.

There are two principal reasons why the specification of an upper limit for crystal activity has not been used more widely. One of these is that for the crystal's position in the oscillator-circuit, particularly when used in the common parallel resonance connections, the saturation characteristics of the vacuum tube tend to alleviate by compensation some of the results of excessive crystal activity. Also, the crystal manufacturing problem is a more difficult one with the additional tolerance in the specification unless predimensioning methods are used. With those crystal units where close tolerances on the impedance (activity) characteristics are part of the design, the using equipment is in a position to incorporate features into its own design which take advantage of a more precisely defined crystal unit.

Following a development by Fair, it is becoming the practice in the study of the performance of the crystal units, if not yet in specifying them, to use the performance index (PI) of a crystal unit as a measure of its intrinsic activity properties. Activity, as it has been commonly used, refers to the d.c. voltage developed in the grid circuit of a vacuum tube when it is oscillating under crystal control. More active crystals develop larger grid voltages. Activity, when defined in terms of oscillator-circuit voltages, is unfortunately dependent upon both crystal properties and oscillator-circuit properties and varies greatly with the vacuum tube and the line voltages used. The activity of a crystal in any given oscillator-circuit is proportional to the impedance of the crystal when in parallel resonance with the circuit load at the oscillating frequency. This impedance, in ohms, is taken by Fair for his PI and is the crystal's contribution to the so-called activity in any parallel-resonant oscillator. In general, this performance index has its smallest values when the crystal is connected across the largest values of circuit capacitance. Values of PI run from a million ohms or more down to a few thousand ohms, a large part of this spread being required for the range of circuit capacitances alone. The PI is proportional to the Q of the crystal, among other factors. The predimensioning method of manufacture is capable of holding the PI and the Q for crystals of any one frequency to within a variation of a few per cent over the entire production.

I. *Voltage and Power Dissipation*

Not only does the high-frequency voltage which the oscillator applies across the crystal depend upon the circuit and upon the vacuum tube which is used, but in the parallel-resonant connection, at least, this voltage is approximately proportional to the activity of the crystal, or to its PI. Thus, a crystal just active enough to pass the minimum specification may in its intended oscillator be driven at, say, twenty volts, while in the same circuit connection a crystal four times as active would develop seventy-five or eighty volts. When circuits using different tubes and placing different capacitances across the crystal are considered, the voltages which are placed across crystals range from ten to one hundred fifty volts.

Whereas the voltage itself is probably not important, except as excessive voltages cause glow discharges and arcing between the crystal surfaces and electrodes, or between the electrodes themselves, the heating of the crystal in excessive vibration sets a severe limitation on the frequency tolerances attainable, as already stated. The power dissipated within the crystal plate or at points of friction in its mounting is given by the ratio of the square of the voltage to the PI in ohms and is expressed in watts. Among the types of crystals which are most common in military communication equipment this power dissipation varies between 1/20 and 1/2 watt. In the development of crystal units to closer frequency tolerances, it will be necessary to keep the power dissipation within the crystal unit to a low figure, perhaps well below one hundred milliwatts. It will require either the use of circuits which do not develop large voltages across the more active crystals, or the specification of an upper limit of crystal activity in manufacture, and/or the provision of means for more ready conduction of the heat developed within the crystal out to the ambient. A rather loose upper limit on activity has already been in effect in the trend to the use of smaller quartz plates without a corresponding change in the minimum activity requirements.

Much of what has been included in the last two sections refers principally to high frequency crystals of military communication equipment used in the parallel-resonant connection. In their series-resonant use, the voltages across equivalent crystals would be very small indeed. The series-resonant resistance of half-inch square pressure mounted crystals in the five to ten megacycle range varies from five to perhaps twenty ohms. Variation of this resistance inversely proportional to the square of the frequency of the plate is inherent for plates whose vibrations are unconstrained. For plates lower than 5 mc/sec, where owing to their extreme thickness clamping at the corners seriously interferes with the freedom of vibration, resistances considerably in excess of the values expected from this inverse square law are found. The activity property of a series-resonant crystal is often described in the terms of its series resistance, and as the latter is made smaller, the crystal is more active. This is in contrast to the association of greater activity in parallel-resonant crystals with their higher equivalent series resistance at parallel resonance.

In applications of crystals as circuit elements at frequencies below 500 kc/sec, there has been much success in controlling both crystal parameters and the properties of the using circuit to a relatively high degree of precision. In this field the total number of units produced is almost insignificant in comparison with the numbers of higher frequency crystals used in military communication equipment, and there has been greater detailed study of the performance and limitations for their more exacting use. Furthermore, precision measurements of crystal properties are easier to make and crystal vibration modes easier to control and predict at the lower frequencies. Comparable measuring instruments and techniques are only just now beginning to appear for the determination of the properties of high frequency crystals, and great progress in this field is anticipated over the next few years.

J. *Finish, Surface, and Mounting of Quartz Resonators*

In the early experience with crystals for military communication, crystal surfaces were not etched after grinding, or at least not etched to a sufficient depth, and a characteristic ageing of crystal units ensued. Under the effects principally of water vapor, but probably also of temperature changes and vibration, the deterioration of an abraded surface was found to proceed to such extremes that the thinner crystals would lose a significant part of their quartz and thus depart from their intended frequency. This surface disintegration progresses over hours or years, depending largely on humidity conditions, and it so limits the stability obtainable from ground crystal surfaces that the practice of finishing high frequency crystals by etching to frequency is now in almost universal use.

Many crystals are plated, the plating providing electrodes in close proximity to the quartz itself and making the full voltage applied by the circuit across the electrodes effective in driving the quartz without a partial loss in an air gap. The direct effect is to provide a considerable increase in activity beyond that which the same crystal would have if not plated. Unless the plating is made thick, it introduces no evident damping and thus does not lower the Q of the crystal. Electrodes so plated directly onto the crystal make possible wire connections which may also serve as mountings for the crystal. One advantage in the wire-and-plating type of support over the more common pressure mounting is that with the soldering of the connections neither jarring nor time disturb the validity of the electrical connection or change either the points of support or the applied constraint, both of which changes are troublesome in pressure mounted crystals. A shortcoming of the former type of mounting is the failure to provide a heat reservoir of large thermal capacity close enough to the quartz plate to receive by an easy conduction path the heat there generated. Such crystals run considerably hotter for the same activity and in the same circuit than do pressure mounted crystals for which thermal conduction across a very short air gap is fairly effective in preventing an excessive temperature rise of the quartz plate itself.

For neither the pressure mounting which clamps the corners of the plate nor the wire suspension type wire mounting with attachments at two peripheral points is the mechanism of restraint and its influence on the mode of vibration of thickness-shear crystals completely understood or very well controlled. An ideal type of mounting for such nodes would appear to be the support of the plate at a central nodal plane with the edges of the plate perhaps tapered to a supporting knife edge. Such mounting arrangements have been tried but are not in common use.

Less constraint is applied to a crystal in the so-called space mounting. Here the plate rests on one electrode while the second electrode is not quite touching. In the case of the thickness-shear mode, which is the principal type under discussion, the plate is obviously constrained to a degree so long as it rests on one face. As the amplitude is increased the plate would be expected first to make only chattering contact and then at larger amplitude still looser contact with the electrode on which it rests, and in this condition be under less constraint with a slight consequent fall in frequency. In making thickness-shear crystals in the half-inch square size at frequencies in the one to three megacycle range, it is becoming common to use spacer mountings in order that the crystal may develop reasonable activity at these frequencies. The restraining effects of corner clamping in limiting the freedom of vibration at the central area of a plate are much more serious for thick plates than for thin.

K. *Holders*

Crystals of the highest precision are contained in hermetically sealed holders which are commonly evacuated to reduce the supersonic radiation losses of energy from the crystal. The development of comparable mountings for military communication crystals has not kept pace with that for precision crystals and filters.

The common phenolic holder has been the source of a good deal of difficulty in several ways. The phenolics have often been permeable to water-vapor so that the humidity within the holder cavity becomes comparable to the humidity of the ambient after a short exposure even though the neoprene gasket used provides a tight seal. With repeated temperature and humidity cycling, drops of water accumulate within the cavity through a pumping action which results from differences in humidity and temperature between cavity and ambient. Again, particularly at the higher temperatures, vapors released by the phenolic either cause corrosion of metal parts within the holder cavity or are available to condense on the quartz itself when the crystal is cooler than the phenolic. The frequency of the crystal is so sensitive to condensation upon its surfaces that fractions of a microgram produce detectable changes in frequency for the thinner crystal plates as well as introducing damping to interfere with the activity.

Some types of phenolics introduce dielectric losses between the terminals or the pins of the holder, particularly after absorbing water, so that the normally high performance index of a crystal suffers a reduction in its ohmic value due to the shunting action of the poor phenolic dielectric. In some equipments even the d.c. leakage of the phenolic has proved troublesome.

Following the realization of the poor performance of the grades of phenolics formerly in use, much improvement has been found in the use of grades less permeable to water vapor which maintain their electrical resistance and which are free from corrosive vapors.

Despite the improved character of the holders now in use, it is believed desirable to use hermetically sealed glass, metal or ceramic holders for communication crystals, particularly with the thought that the natural trend in crystal control will be toward crystals meeting closer frequency tolerances. The misbehavior of the holders has been one of the principal obstacles in the attainment during a long life of the ±0.02 per cent frequency tolerance over the wide temperature range which many military crystals must meet. In the development of hermetically sealed holders the problem is, in part, to find ways to obtain a seal which is equal to that used in vacuum tube technique while still adaptable to present form factors. There is no desire at the present time to have an evacuated holder for these crystals, but almost complete absence of water and other vaporizable materials which might deposit on the crystal to shift its frequency is regarded as an essential. It is the present view that the most likely practical test of tightness of the hermetic seal is to immerse the sealed holder in saturated water vapor directly following long exposure in a vacuum chamber, the crystal itself to serve as an indicator of the admission of water vapor to the cavity.

VI. A Comment on the Future of Piezoelectric Resonators

There are no alternative sources in sight for the enormous values of Q which quartz makes possible in resonators. Thus it would appear that any needs for the utmost in frequency precision and stability will continue to be met by the use of quartz.

There are large areas of the frequency control problem where the ultimate in constancy of frequency is not required and where the choice of a piezoelectric resonator instead of a wire circuit is to some extent one of convenience, cost or space, and where the required performance could be obtained through appropriate sacrifices without the use of quartz. It may continue to be the practice to use quartz in such cases although not pushing its control features to the limit. Much might depend on the continued availability of the supply of quartz. The degree of frequency constancy needed in many applications could probably be obtained using very much smaller pieces of quartz, but difficulties in handling during fabrication and the inherent greater percentage of waste in sawing and lapping tiny pieces are definite obstacles. It is in uses where the highest Q's are not required that some mechanically inferior substitute for natural quartz might well offer the convenience of mechanical frequency control while admittedly providing a degree of frequency constancy lower than that which quartz might provide.

While the convenience of the quartz resonator in portable and mobile radio equipment lies partly in the freedom from temperature control which the peculiar elastic properties of quartz make possible, many of the advantages of crystal control would persist despite the possible necessity for thermostating a substitute crystal if it were developed. In view of the growing practice of mounting in a hermetically sealed holder and shock mounting the crystal on wire suspensions, a new piezoelectric crystal need not necessarily meet the high mechanical standards of quartz.

Quartz itself, particularly as it pioneers in new fields of more extreme frequency precision, or into higher frequency ranges, will probably show that its full powers in these directions have been far from being exploited. To mention just one factor, the problem of the surface stability of quartz which was not considered in its full light until its appearance in the recent experience with ageing units, is well on its way toward clearing the road to the use of thinner and higher frequency crystals; and this same stabilizing of the surface is one of the factors in the attainment of the highest Q's, the extreme values of which are not yet fully utilized.

The existence of the simple and the elaborate designs of crystal units in today's manufacture emphasizes again the two extremes of requirements noted earlier in the comparison between low and high Q materials in their future applications. On the one hand, quartz units made, mounted and sealed with precision according to a well worked out design, give a performance which is consistent to an extreme degree, and with the minimum in frequency tolerances. The use of precision units is amply justified when the application calls for these extreme tolerances, for there is no other way to achieve the required end than by the use of quartz and by the close attention to the great detail in its fabrication and in its adaptation which is required. On the other hand, crystal units at the other extreme in the perfection scale have their proper application as well. For this type of unit it has been amply demonstrated during the past few years that there can be obtained in enormous volume at ridiculously small cost, a unit which is highly useful, and whose properties meet tolerances which, though at least one order less rigid than in units of the precision class among crystals, make it nevertheless a precision device in comparison with other circuit elements.

The systematic development of units in this latter field, however, despite the great progress which has been made, has not received the attention which it deserves. This is largely because of the pressure for production, its enormous expansion under wartime demands, and the necessity for emphasis upon correcting existing designs and upon controlling the quality of the product. The time is now believed to be ripe for studies of the basic circuit properties and circuit behavior of crystals in this latter field, along with the re-evaluation of the designs, and for the development of instruments and measuring techniques for these new components of high frequency circuits. Research and development work in this field is now in a position to capitalize on extensive practical experience which is a guide that was largely lacking a few years ago. In the view of the writer, the high frequency crystal unit is approaching the stage in its evolution where it will become as well standardized and as well measured and specified a circuit component as are resistors, condensers, coils and tubes today. Furthermore, its properties will be similarly stated in terms of conventional electrical units. The reduction of performance to standard electrical engineering terminology, the refinements of design and of processing controls to assure meeting the circuit values found to be desirable and practicable, and the development of a circuit formulary and instrumentation for this new branch of radio engineering represent, however, projects which will require several years for their accomplishment.

VII. Listings of a Few References in the General Field of This Paper

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GEOLOGY OF QUARTZ CRYSTAL DEPOSITS

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Introduction

Quartz crystal was, before the war, a minor nonmetallic resource. Wartime demand for large-sized clear single crystals of quartz for use in making quartz oscillator plates for radio frequency control has emphasized the strategic importance of this industrial mineral. Quartz crystal of usable quality is of limited occurrence, and little has been written regarding the geology of the deposits although there has been considerable work done during the war period. There are also interesting relationships to be brought out between the mineralogical and geological characteristics of quartz and the problems inherent in the supply and use of this commodity. It is the purpose of this paper to discuss the geology and mineralogy of quartz crystal deposits and to point out the interdependence of quartz crystal geology and the supply situation in Brazil.

The information contained in this article has been gathered during more than three years' work by each of the atuhors in the government service on problems involving quartz crystals. Geological description is in part the result of the authors' work but is chiefly compiled from unpublished work of others. Most important is the work of the geologists and mining engineers employed in Brazil by the Foreign Economic Administration, to whom acknowledgment is particularly made. Unpublished information of the U. S. Geological Survey on domestic deposits has not been included, but publication by that agency is expected in due course. Much of the geological information on quartz deposits throughout the world remains to be correlated. Some is still restricted. The interim observations in this paper may, however, serve as an introduction to an understanding of the geology of this commodity and the relationship of the geology to the problem of supply.

Size and Quality Standards and their Effect on Supply

The minimum size and quality requirements for quartz crystal to be economically usable are somewhat indefinite and depend upon the manufacturing method to be used, changing economic factors such as price, and to some degree upon the preference of the user. Individual crystals are required to be approximately 100 grams (about 4 oz.) to 2000 grams (about $4\frac{1}{2}$ lbs.) in weight; shape is a factor in material less than 200 grams in weight. Crystals both larger and smaller find some use in the oscillator plate industry. The former are difficult to saw and are high priced; the latter require more labor in their use and will usually not allow fabrication of the $\frac{3}{4}''$ square or 1″ square plates. Industry has accommodated itself to the use of unfaced crystals above 200 grams in weight. The quality of the stone is also a fundamental factor. Many types of defects are considered in classifying radio grade crystal. The clarity of the stone determines whether it meets the minimum acceptable quality levels. Generally 50 per cent of each stone must be free from any visible defects when the stone is suitably illuminated. Usable crystals are further subdivided on the basis of additional tests. If less than one-half the volume is eyeclean, use of the stone is generally uneconomic. National Bureau of Standards specifications are slightly higher.

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These requirements both as to size and quality, as stated, are considerably different from the acceptance standards in use before the war. In general, stones less than 2 lbs. in weight did not find ready acceptance and the presence of several natural faces was usually required; various minor imperfections now allowable were considered to render crystals unusable. Of the companies manufacturing oscillator plates, only one of the twenty-eight which reported in February 1942 indicated that the quartz crystals they used averaged less than 1 lb. in weight. This company used crystals averaging $\frac{3}{4}$ of a pound. Nineteen of the twenty-eight companies reported that 1 lb. was the minimum size. In the earlier days of the war the trend toward the use of smaller-sized stones began and by early 1943 their use was general throughout the industry. It is estimated that in February 1942, 25% of the consumption was of crystals from 200 to 500 grams in weight, and 75% of the consumption was of crystals 500 grams and heavier. In 1944, 30% of the consumption was estimated to be less than 200 grams, 50% to be 200 to 500 grams and only 20% to be 500 grams and higher. There was considerable emphasis on minor defects during 1942 due to desire of the manufacturers not to jeopardize badly-needed production by using raw material of questionable quality. However, in 1943, with the advent of more efficient manufacturing techniques, larger plant capacity and increased demand for raw material, the defects were widely accepted and successfully utilized. Quality standards were relaxed somewhat to the present minimum standards which have been described. The development of the use of smaller sizes and lower quality crystals was of great importance in connection with supply in that it rendered usable large quantities of hitherto rejected material.

Supply of Quartz Crystal

The supply of quartz crystal which is used in the U. S. comes almost entirely from Brazil. Deposits are known and worked in USSR[1] and small deposits have been investigated elsewhere. Some quartz has been used by the U. S. industry which has been mined in the states of Arkansas, Virginia, North Carolina and California, and a small amount of material has been imported from Guatemala, Colombia and USSR. Since the production has been very small from sources other than Brazil, it has had no appreciable effect on the overall United States supply situation.

U. S. imports of Brazilian quartz for consumption in 1939 were 67,052 lbs.; in 1940, 126,521 lbs.[2] Imports since 1940 are measured in hundreds of tons rather than in thousands of pounds. The problem of insuring availability of quartz crystal for necessary war production has received the cooperation of private industry and government throughout the war period both in U. S. and Brazil.

Buying of quartz was instituted in Brazil by the U. S. Government in 1941, and production has been reserved to the United Nations. U. S. Government purchases in Brazil have been under the successive auspices of Metals Reserve Company and the U. S. Commercial Company, a subsidiary of Foreign Economic Administration. Government-purchased quartz has been sent to the National Bureau of Standards, graded and placed in the national stockpile in custody of the Metals Reserve Company. Quartz is released by the War Production Board from the stockpile to consumers when justified and is used for the manufacture of quartz crystal units and other strategic items for the armed services. Private importers who were actively engaged in importation of quartz before April 1942 have continued in business under WPB regulations and have provided a large part of the quartz imports used for these strategic items during the war period. This quartz is supplied direct to consumers. The grading practice employed by the importers varies somewhat from that of the Bureau of Standards, but the variation in the standards of quality required by industry due to differences in processing methods have served to allow quartz graded by both NBS and the private importers to find an appropriate market.

Geology of Quartz Deposits

The geology of quartz deposits in various parts of the world is only beginning to become known. Such material as is available has in most cases not yet been correlated due to pressure of war work. In the discussion which follows, the description of deposits and general observations on the geology and mineralogy are made with the realization that studies will become available which will provide more detailed information.

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[1] *Foreign Commerce Weekly*, **15**, No. 6, p. 28 (1944).

[2] *Minerals Yearbook*, Review of 1940, U. S. Bureau of Mines, p. 1422 (1941).

Quartz in Australia

Productive quartz deposits in Australia are located in northern New South Wales at the Kingsgate mining camp, twenty miles from Glen Innis. Crystals of various sizes have been produced and used by Australian oscillator plate manufacturers but the production has not met Australian requirements.

The mines in this camp were worked for molybdenite and bismuth in the last war. During the present war, some of the mines have been reopened for quartz crystals. The mineralized area in which quartz occurs has been worked over a zone $1\frac{3}{4}$ miles long and $\frac{1}{2}$ mile wide, which is within a granite near the contact with slate.[3] Here there are scores of pipes three to sixty feet in diameter. These pipes plunge in all directions and some of them have been shown to extend to depths of 500 feet. Some pipes are bordered by slate along part of their extension in depth but none is entirely within the slate. Molybdenite, wolframite and native bismuth as well as carbonate and oxides of bismuth are present and gold and silver are associated with the bismuth. Within the pipes or in branches or bulges which are tributary to them, are open cavities lined with quartz. In some cases the crystals occur on the hanging wall side. Less than 1 lb. of usable quartz per ton of rock mined was reported from various operations over a period of several months. The occurrence stands out as the only described example of usable crystals in a deposit economically productive of metallic minerals.

Quartz in Guatemala

Guatemala quartz deposits are located[4] in the Department of Baja Verapaz, north of Guatemala City and occur in an area of gneisses and schists. The occurrences are characteristically small gash veins in the schists. The veins contain fine-grained quartz at the border and well-formed crystals in the clay material which occupies the central part of the veins. The crystals in the few tons of radio grade quartz produced have been largely 200–500 grams in weight.

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[3] Van Wagenen, H. R., Unpublished FEA Reports (1943).

[4] Schmidt, W., Houck, J. G., and Irving, Earl M., Unpublished FEA Reports (1943 and 1944).

Quartz in Colombia

A productive quartz deposit has been developed in Colombia near the town of Muzo, Boyaca, Colombia.[5] It consists of one principal vein and numerous subordinate veins. These veins are localized by fractures in a drag fold structure on the vertical limb of a tight anticline in the Cretaceous slates (Villeta formation). Milky fractured comb quartz fills the main vein which averages about 3½ feet thick and dips 30 degrees. Iron oxide and calcite are the only associated minerals reported. Significant amounts of usable crystals persisted to only a few meters in depth. The quartz crystal is well terminated at one end and the clear crystals line irregular cavities. The cloudy base of the crystals necessitated extensive trimming. A few tons of merchantable crystals largely 200 grams to 500 grams in weight have been produced, although a few crystals weighed up to 13 kilograms.

Quartz in Madagascar

Before the war some quartz was imported from Madagascar and used for the manufacture of oscillator plates in this country. Although Madagascar quartz had a reputation of poor quality, it appears to have been as good or better than the quartz now in use in the oscillator plate industry. The most productive deposit in bedrock is reported to be some 200 miles south of Tananarive. Usable quartz occurs in well-formed crystals, lining vugs in veins of milky quartz. No other minerals are reported from the deposit. Alluvial deposits are reported from several localities; one of these is near Vohemar near the northeast end of the island. Scattered information suggests that the production from Madagascar consists of a somewhat larger proportion of larger-sized crystals than the Brazilian production.

Quartz in the United States

The search for radio quality quartz in the U. S. during the war period has been stimulated by the War Production Board, Metals Reserve Company, the U. S. Geological Survey and numerous private concerns. As a result of these efforts a good deal more than formerly is known of the occurrence of radio grade quartz in the U. S. Information regarding the localities in the U. S. has been gathered and is being correlated by the U. S. Geological Survey. The remarks included in this paper serve to mention only a few points of interest regarding quartz in this country.

The greatest number of usable crystals has come from Arkansas, California, western North Carolina and southwestern Virginia. The Arkansas deposits have recently been described and preliminary information about other deposits in U. S. has been released.[6] Crystals of good quality have been purchased in Arkansas but no mines have been worked much beyond the development stage, largely because of the cost involved. In several of the prospects which were worked, crystals were mostly in the smaller weight class; in one other the crystals were characterized by excessive electrical twinning.

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[5] Burns, W., Singewald, Quentin D., and Leland, G. R., Unpublished FEA Reports (1943 and 1944).

Crystals have been produced in southwestern Virginia and western North Carolina largely as a result of activity stimulated by the United States Government. With one or two exceptions the crystal has not been found in place in veins but has been found in the weathered mantle on the farms of the region. The crystals are in general larger than 200 grams. In the same localities where the crystals have been found, veins of massive quartz outcrop. So far as is known these veins have not been explored except by one or two prospect pits. The genetic connection between the outcropping veins and the crystals in the soil has not been established.

Most of the quartz produced in California has come from buried placers in the Sierras. Individual crystals are of very large size but available information does not suggest that any large amount of crystal is to be expected from this source.

In general, the widespread search and examination of reported occurrences of radio grade quartz in the U. S. has indicated areas of some promise as has been mentioned above. In California and in Arkansas where the greatest amount of exploration has been carried on, the results suggest that there is no large tonnage available. From the results obtained, it appears that production from domestic sources could not be great enough to contribute significantly to the requirements such as were demanded by this war.

GEOGRAPHICAL DISTRIBUTION OF QUARTZ DEPOSITS IN BRAZIL

The principal quartz deposits of Brazil are situated in the states of Minas Gerais, Goiaz and Baia. So far as is known none of the other states yield significant quantities except Espirito Santo and Para. Quartz of good quality is occasionally reported from Paraiba, Matto Grosso, Maranhao, Amazonas, Pernambuco and Ceará. Almost no quartz crystal occurs south of the latitude of Rio de Janeiro.

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[6] Miser, Hugh D., Quartz veins in the Ouachita Mountains of Arkansas and Oklahoma: *Econ. Geol.*, **38,** 91–118 (1943). Engel A. E. J. and others, Geological investigations of U. S. quartz crystal deposits with special reference to those of Arkansas: Presented at A.A.A.S. Section E Meeting Sept. 1944. Quartz crystal deposits of western Arkansas: *Dept. of Interior Information Service*, Nov. 9, 1943 (announces availability of maps of the main quartz-bearing areas and individual deposits). The Rough Diamond Crystal Mine and the Calaveras Crystal Mine, Calaveras Co., Calif.: *Dept. of Interior Information Service*, Sept. 4, 1943.

The relative production of the various Brazilian states has been estimated as follows: Minas Gerais 35%, Goiaz 30%, Baia 20%, Espirito Santo 5%, all others 10%.

Minas Gerais

About three-fourths and perhaps more of the production of Minas Gerais is realized from a zone beginning just north of Belo Horizonte and extending northward to the Serra de Minas and Serra do Cabral (Fig. 1). The zone of producing areas is about 250 kilometers wide as there are deposits near Pitangui, west of Belo Horizonte and at Itamarandiba, northeast of Diamantina. The trend of this zone coincides with the railroad line north of Belo Horizonte, and it may be that eastward and northeastward extensions are present but unexploited. Promising occurrences have been reported to the north in the valley of the Rio Jequithinhonha,[7] but these are comparatively undeveloped due to their geographical remoteness. The most important center of production is around Sete Lagôas but Montes Claros, Curvelo and Diamantina to the north within this same productive zone are also prominent centers. Crystal from central Minas Gerais has a reputation for good quality. Some of the occurrences in this state have been described by Kerr and Erichsen.[8]

Goiaz

Approximately half the production from Goiaz is mined near Pium in the hills west of the Rio Tocantins in the north central part of the state (Fig. 1). The nearest center is Porto Nacional. At least one-quarter and perhaps one-third of the production of the state originates in southeastern Goiaz near the town Cristalina. Anapolis, and Ipameri, approximately 125 kilometers further south, are the principal shipping points for all Goiaz crystal. Other producing areas are present in east central Goiaz and in the northern and western parts of the state.

Baia

The most important producing district in Baia is located in the north central part of the state, east and south of the Rio São Francisco, southwest of Joazeiro on the river, and has been designated as the Mimoso-Alegre-Batateiras district (Fig. 1).

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[7] Johnson Fred E., Unpublished FEA Report (1943).

[8] Kerr, P. F. and Erichsen, A. I., Origin of the Quartz Deposit at Fazenda Pacu, Brazil: *Am. Mineral.*, **25**, 487–499 (1940). Bibliography of earlier papers on quartz crystal in Brazil is included.

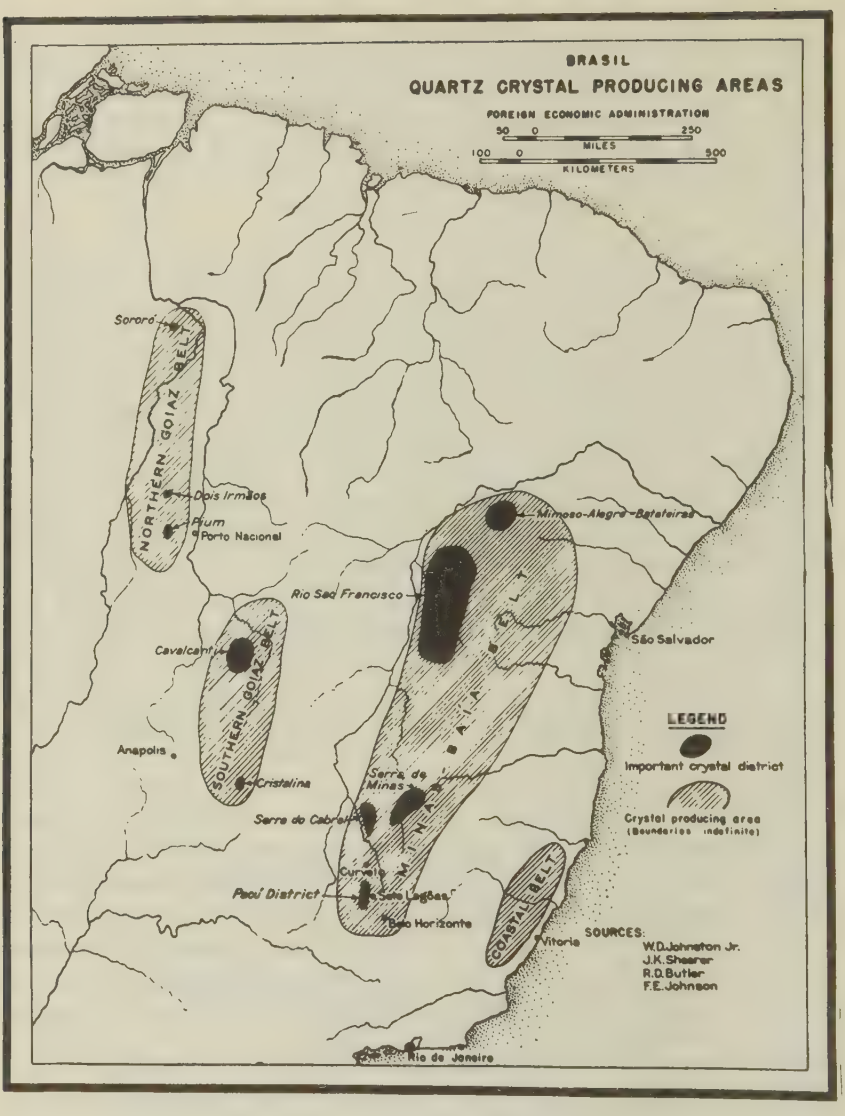

FIG. 1

The other producing region of importance lies in the central part of the state and is virtually continuous with the north central area. Various centers of production have been reported from the southern and southeastern parts of the state, for example Conquista, from which excellent quartz has been obtained. In general the districts in Baia are situated east of the Rio São Francisco. Information is extremely scanty regarding the area between the producing areas in Baia and those to the south in Minas Gerais. This region is most inaccessible and may contain important deposits of crystal not yet developed.

Espirito Santo

Numerous small deposits occur in the central part of the state near Vitoria and south of the Rio Doce.

Summary

The regions in Brazil which produce quartz lie in four belts or zones which trend north to northeast in direction as shown in Fig. 1. These belts are: (1) Northern Goiaz—extending for at least 600 kilometers in the northwestern part of the state, west of the Rio Tocantins; (2) Southern Goiaz—extending from the vicinity of Cristalina, Goiaz, northwards along the Goiaz-Minas and Goiaz-Baia border for 500 kilometers with ultimate northern extremity not known; (3) Minas-Baia—extending some 1200 kilometers from Pitangui and Sete Lagôas in Minas Gerais to the vicinity of Sento Sé and Joazeiro on the Rio São Francisco in Baia; and (4) Coastal—a belt some 300 kilometers in length, including the deposits in Espirito Santo, and those near Aimores, Minas Gerais, at the northern end of the belt. The northern Goiaz zone continues into the adjacent state of Para.

TYPES OF GEOLOGIC OCCURRENCES IN BRAZIL

For convenience of description the Brazilian quartz deposits may be classified into the following types:

1. Veins and composite lodes
2. Pipes and pockets
3. Blanket deposits
4. Pegmatites
5. Eluvial and alluvial deposits

Deposits of the first three classes mentioned are gradational one to the other. The striking feature of all the deposits is the extremely small proportion of usable crystal in relation to the total quartz. Enormous quantities of milky bull quartz are mined to produce a few kilograms of usable clear crystal. The production comes from hundreds of mines or diggings, very few of which involve underground mining. It is estimated that during the war period there have been no more than six to twelve mines in Brazil in which there has been production of one ton a month for any length of time. Most diggings produce less than 500 pounds per month and many only 50 or less.

Veins and Composite Lodes

Veins and composite lodes yielding commercial quartz are a particularly important type of deposit in the southern part of the Minas-Baia belt. Wall rocks are commonly shale or shale and granite. In other parts of Brazil veins occur in quartzite or sandstone although in these rocks the yield from accompanying bedded replacements, pipes or pockets is the more important.

In the shale near Sete Lagôas in Minas Gerais lodes are fairly persistent along their strike, although in most instances individual shoots or lenses within the veins are not so persistent. The quartz commonly forms conformable bedded deposits in the walls of the vein proper. Because of the varying nature of the crystal occurrences in the walls as well as the variation in width of the lode or vein system, mining widths may show a wide range in any one deposit.

Veins, lodes and their accompanying bedded deposits contain milky bull quartz, clay, manganese and iron oxides, and remnants of country rock. Clear crystal occurs in groups or individuals embedded in the bull quartz or in places in vugs usually nearly filled with material. The clear quartz appears to have formed at a later stage than the bull quartz. Manganese oxide is more abundant in the vicinity of crystal and usually can be considered as an indicator. This association does not assume genetic significance as the time of formation of the crystal and the oxide could have differed greatly.

The best example of a lode in shale is furnished by the Guachi mine near Sete Lagôas (Fig. 2). The vein system has an approximate north-south strike and moderate westerly dip averaging about 60 degrees. Mining has occurred along a strike length of 400 meters and to a depth of some 25 meters below the original surface. The wall rock is weathered shale, yellow in color, lying immediately below a red clay capping which contained some crystal. The system is composed of a series of lenses which pinch out both vertically and horizontally. Greatest mining width reached was a few meters, but horses of country rock occupied much of the cross section. The distribution of values was erratic. The workings at the Guachi mine have now been abandoned because with depth the yield of crystals became uneconomic.

An excellent example of a vein system occurring along a shale-granite contact is afforded by the Onca Mine, also in the Sete Lagôas District. This has been one of the most productive mines with a steady production of one to two tons of mine clean quartz per month. Fig. 3 illustrates the main vein workings, and Fig. 4 represents a geologic cross section of the deposit. The strike length exposed on the mine workings is 500 meters. The workings reach a depth of 30 meters beneath the original surface. The vein strikes approximately north-south and dips to the west at moderate angles ranging from 45 degrees to 65 degrees. The rock on the east (footwall) side of the vein is weathered granite, on the west (hanging wall) side is weathered shale. The lode is composed of a system

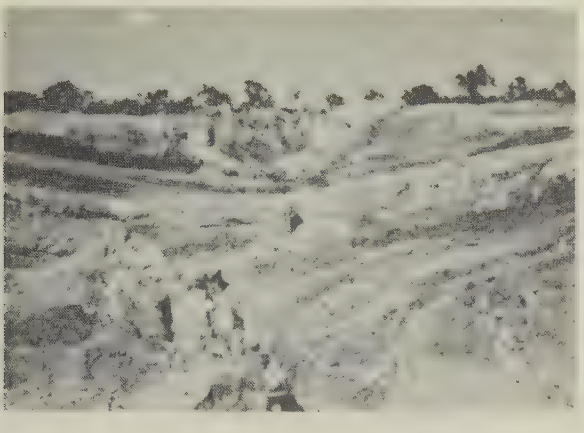

FIG. 2. Looking south along the strike of the vein system at the Guachi mine, near Sete Lagôas, Minas Gerais. Mining of nearly vertical vein by hand methods in foreground. Red overburden has been stripped off and mining will advance to center of picture. Old workings are in background pit on same vein zone although individual veins pinch out along the strike.

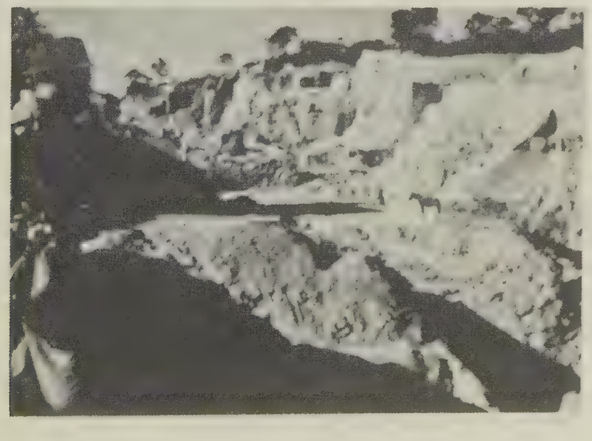

FIG. 3. Looking south along strike of the vein zone, Onca Mine, Sete Lagôas, Minas Gerais. Weathered shale to the right is the hanging wall. Footwall of granite is in shadow to the left.

of quartz stringers, veins, and lenses occupying a position in the shale adjacent to the granite. Horses of shale are common. Along the contact and decreasing away from it into the hanging wall, the shale is somewhat folded and distorted with extremely variable dips. It appears in general to have moderate dips, slightly flatter than the contact dip near the contact, and to become flatter to the west. Quartz also occurs along the bedding planes in the crumpled shales of the hanging wall (Fig. 5).

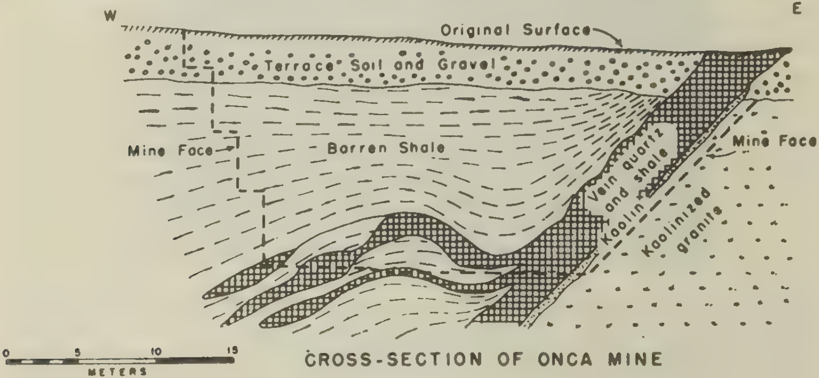

FIG. 4. Cross section of Onca Mine. After H. K. Shearer.

Due to the structure of the lode and the varying quantity of hanging wall quartz, mining widths vary greatly, ranging up to 20 meters in places. It is held that the granite has intruded the shale, or the shale has been faulted against the granite, or the shale was deposited adjacent to an eroded granite ridge. H. K. Shearer who has done the most work in the region advocates the depositional contact.

The important deposits near Pium, Goiaz, as described by H. K. Shearer[9] are best classed as veins. Wall rocks are schists of sedimentary derivation and intercalated quartzite. Weathering has rendered the formations soft. The quartz occurs in irregular veins and lenses, transecting the schist and quartzite. Individual crystals of bull quartz attained enormous sizes. Shearer mentions one 2.5 meters in length and 1.5 meters in diameter. Clear crystal of commercial value represents only a fraction of the material which is mined.

Pipes and Pockets

Pipes or irregular conical or funnel-shaped masses and pockets of quartz occur in Baia, southeastern Goiaz, Minas Gerais and perhaps elsewhere. Wall rocks are quartzite or sandstone. The sizes of the pipes and pockets show great variations, and accompanying veins, pods, bedded replacements, and lenses are common. Some deposits which are classed with pipes are more in the nature of elongated pods. There is little essential difference between a pipe, pocket or the common blanket deposit in quartzite or sandstone. Some of the deposits in the vicinity of Cristalina, Goiaz, include all variations. Zones of small pockets, veinlets and replacement masses lie in sandstone along a north trending structural axis. The deposits in the Cristalina district have been described by W. D. Johnston Jr.[10]

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[9] Shearer, H. K., Crystal Producing Areas of Northern Goiaz and Southeastern Pará: Unpublished FEA Report (1943).

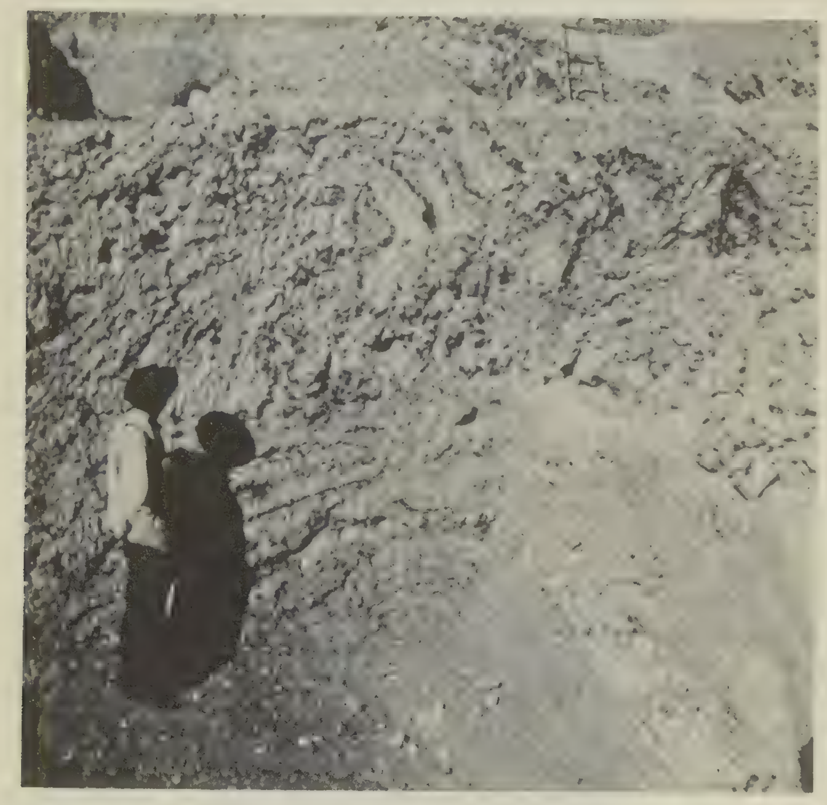

FIG. 5. Official U. S. Navy Photograph. Veins in folded shale of the footwall, Onca Mine, Sete Lagôas, Minas Gerais. Veins contain quartz and manganese oxide. Similar veins, somewhat larger than most of those in the picture are the source of much good crystal.

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[10] Johnston, W. D., Jr., Cristal de Rocha em Cristalina, Estado de Goiaz: *Div. fomento produção mineral* (Brazil), Avulso No. 57 (1944).

Perhaps the best example of a pipe-like deposit is afforded by the Alegre mine some 20 kilometers south of Mimoso, Baia. The country rock is compact, fine-grained quartzite. According to F. D. Hanson[11] the pipe-like mass of quartz is localized at the intersection of two fairly prominent groups of closely spaced joints or fissures of small displacement. The bulk of the material in the pipe is coarse milky bull quartz containing some comb quartz and well-formed crystals. The pipe is approximately 10 meters by 15 meters in horizontal dimensions and has been mined to a depth of 15 meters. The walls are extremely irregular and pockets, tongues and masses of quartz protrude from the margins into the surrounding rock. Clear crystals occur in irregularly shaped pockets within the bull quartz throughout the volume which has been opened. There is no clue to the structural distribution of the valuable material. Pockets are filled in varying degrees with clear crystal which is well terminated. Replacement of the country rock was the dominant process although the silica may have been provided from the surrounding quartzite.

An interesting and common feature of the crystal from the Alegre Mine is the presence of phantoms of manganese oxides in many of the otherwise clear crystals. Except for this defect, the crystal is reported to be of good quality.

Blanket Deposits[11a]

Blanket replacement deposits are well developed at Mimoso and elsewhere in Baia, at many places in Minas Gerais, and in Goiaz. It is common for this type of deposit to be associated with veins in shale sandstone, or quartzite, and with pipes, pods, and associated forms in sandstones and quartzites. The deposit at Mimoso is not associated with accessory types and furnishes a spectacular example of blanket deposit from which there has been important production.

Mimoso lies about 200 kilometers west of Bomfim, Baia, in the midst of a dissected plateau with sandstone and quartzite exposed at the surface. Shale underlying the sandstone is exposed in the valleys. A broad flat anticlinal dome with its axis trending east-west shows an indefinite terrace on its southern flank. Dips range from 4 degrees to 7 degrees and the terrace is essentially horizontal. Three beds of replacement quartz occupy closely spaced positions in the shale country rock (Figs. 6, 7 and 8). The horizontal continuity of these beds has not been entirely established. The middle bed is relatively insignificant, thin, and restricted in its occurrence and will not be discussed further. All the beds

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[11] Hanson, F. D., Unpublished FEA Report, 1943.

[11a] According to W. D. Johnston Jr., who has recently carried out detailed studies in some of the quartz districts, certain deposits classed as blanket type furnish indications that filling of open spaces by quartz could have been more important than replacement in the formation of the deposit. F. D. Hanson and W. D. Johnston Jr. have in preparation a paper describing the details of the deposit at Mimoso.

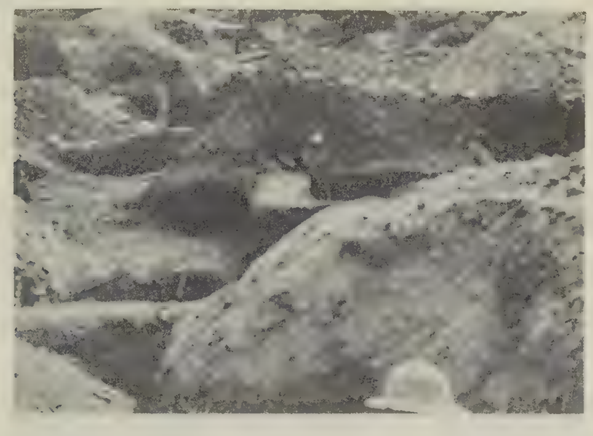

FIG. 6. Pits in shale, some of which extend down to lower bull quartz horizon, Mimoso, Baia. Walls of broken rock between pits are shored up with boulders of bull quartz which have been mined in search for crystal. Stripping has exposed upper quartz horizon in left center of the photograph. Man in center stands on top of upper quartz horizon.

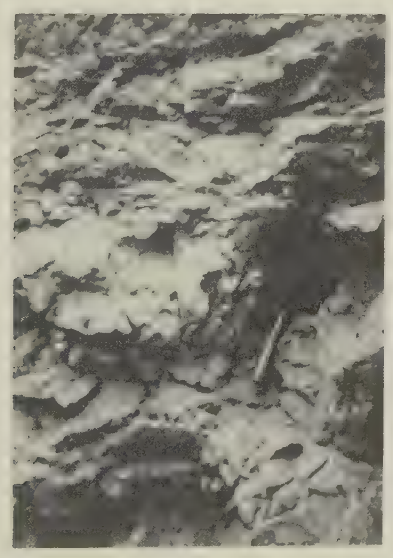

FIG. 7. Blanket deposit. Mimoso, Baia. Coarse-grained bull quartz and pockets from which commercial crystal has been removed.

contain residual masses of shale. Here and there erosion has exposed the beds but for the most part they are covered by partly weathered shale. The upper bed probably underlies an area of about 2.5 square kilometers on the south flank of the dome. It ranges in thickness from less than one to two meters. The lower bed underlies a known area of about 2 square kilometers more or less coextensive with the terrace but extending beyond its limits, particularly to the west and to the south down the dip. It ranges in thickness from about 1 to 4 meters. The average separation between the two beds is about 8 meters. Each bed has produced large quantities of crystal.

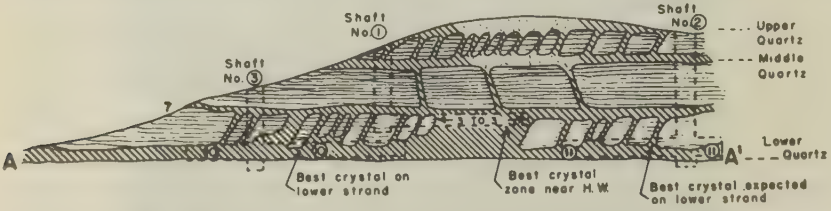
Shaft No.①
Shaft No.②
Shaft No.③
Upper Quartz
Middle Quartz
Lower Quartz
A
A'
?
10
11
11
Best crystal on lower strand
Best crystal zone near H.W.
Best crystal expected on lower strand

FIG. 8. Cross section of the important blanket deposit, Mimoso, Baia. After J. C. Schell. Looking northeast. Horizontal distance represented is 350 meters. Vertical scale exaggerated.

The best production from the upper bed was realized from the central part of the area along a belt parallel to the structural terrace and overlapping its extent towards the crest of the dome. Many parts of the lower bed have been prospected up to this time but the best production has been obtained from a small zone of about 10,000 square meters area, lying in the structural terrace. Small abrupt folds striking northeast-southwest occur in the vicinity of the best crystal (Fig. 9), and tiny quartz-filled joints are present in the shale beds. Prospecting along the trends of folds or groups of joints has been fairly successful. In the outlying part of the district active prospecting along these features has, however, not yet disclosed commercial crystal.

Manganese and iron oxides and clay are the only visible minerals accompanying the quartz. The lower bed, for example, consists of massive bull quartz of varying crystallinity, some of the crystals being fairly well-formed. At planes thought to be localized or at least related to the structural features in the shales, manganese staining becomes more prominent and continued work at times leads to a pocket of crystal. The pockets range from a few centimeters in size to several meters and are filled to varying degrees with well-formed crystal not all of which, however, is a commercial product. The commercial crystal characteristically contains "blue needles." Pockets may be within the massive bull quartz or make out into the roof or bottom. There does not appear to be a favored vertical position within the quartz bed for the development of crystal. Enormous quantities of material have been mined at Mimoso. Much of the work, however, has been in an attempt to reach the crystal zone itself so that the ratio of earth moved to crystal produced has depended upon the depth of the bed.

At the Rosilho mine, southeast of Mimoso, a steeply dipping vein flattens near the surface to form a blanket of small extent in the country rock which is sandstone. Adjacent to the bull quartz blanket the sandstone is noticeably silicified. This occurrence provides a clear example of wall rock alteration adjacent to a quartz deposit. In general, however, observation of the effects of wall rock alteration is difficult due to the extensive weathering.

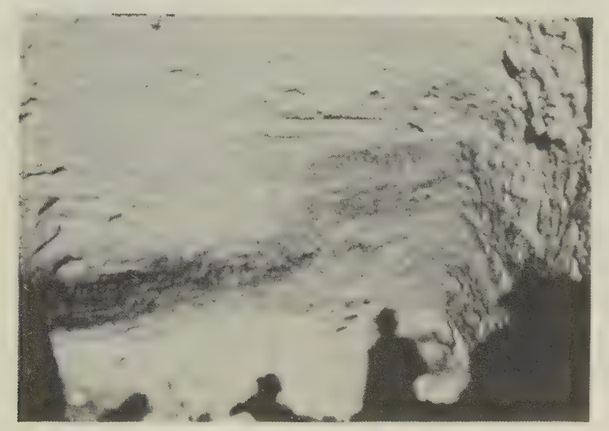

FIG. 9. Small fold in shale. Mimoso, Baia. These folds appear to be related to concentration of crystal in the bull quartz exposed in the bottom of the pit.

Pegmatites

True pegmatites have not contributed appreciably to the quartz production of Brazil. It is suspected that the pegmatite belt of eastern Minas Gerais may contain some crystal, but production from true pegmatites appears to be insignificant. Deposits from Teofilo Ottoni, Minas Gerais, are reported to be of pegmatite type. It is possible that the crystal from the swamp-covered alluvial deposits of Espirito Santo is of pegmatitic derivation as suggested by Knouse.[12] Veins or masses with feldspar could be classed as pegmatitic or possibly as deposits allied to the pegmatites. Two representatives of this type are known: one near Conquista, Baia, the other near Oliveira dos Brejinhos, Baia. The crystal from Conquista is of excellent quality and although material from Oliveira dos Brejinhos is supposed to be good, no first hand data are available.

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[12] Knouse, F. L., Cristal de Rocha no Estado do Espirito Santo: *Div. fomento produção mineral* (Brazil), Avulso No. 55 (1944).

Oliveira dos Brejinhos lies about 60 kilometers southeast of Jardinopolis which is on the Rio São Francisco. The quartz mine is a few kilometers west of the town. According to F. L. Knouse,[13] a formation composed mainly of fine-grained quartzite strikes north-south and dips 60 degrees west from a ridge west of Oliveira dos Brejinhos. Two sill-like masses of quartz and kaolinized feldspar constitute the deposit. One is about 4 meters thick, the other about 2, and they are separated by about 7 meters of country rock quartzite. About 80% of the bodies consists of a quartz core; kaolinized feldspar with tourmaline needles lies along both the foot and hanging walls. Commercial crystal occurs throughout the quartz but particularly so in the kaolinized footwall.

The deposit near Conquista, Baia was examined by D. de C. Smythe.[14] Vertical veins a few centimeters in thickness cut sandstone and gneiss exposed along the slopes of a low hill. The veins have a central zone of quartz and margins of red feldspar. Crystals are large and some of them contain usable portions.

Eluvial and Alluvial Deposits

Eluvial deposits of quartz occur throughout the crystal-producing region of Brazil. They grade on the one hand into alluvial deposits and on the other into primary material in place. The surface zone at Pium may be considered as eluvial, and there are numerous examples in Minas Gerais (Fig. 10). Crystal is mined from many places where eluvial and alluvial processes and primary deposition are all important and it is difficult to diagnose the actual relations. Float at the surface has been the common indicator of underlying deposits (Fig. 11). Quartz from eluvial deposits may show a considerable degree of rounding. It is probable that while some of these deposits may be the eroded roots of primary deposits, others may overlie primary deposits and represent erosion of a considerable thickness of overlying material concentrated by gravity without lateral movement.

Undoubtedly alluvial deposits occur throughout the crystal-producing region, but so far as is known are best represented in central Minas Gerais and Espirito Santo. Present streams occasionally yield commercial stones as a by-product of diamond placering. In general alluvial deposits lie buried beneath clay, particularly red clay, or beneath swampy ground as in Espirito Santo.[15] They are characterized by small-to-medium-sized stones, the commercial ones being of good quality though usually somewhich rounded (Rollados), contained in a relatively thin bed of clay. In places several beds occur separated by a meter or so of noncrystal-bearing clay, sand or gravel.

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[13] Knouse, F. L., Unpublished FEA Report, 1944.

[14] Smythe, D. de C., Unpublished FEA Report, 1943.

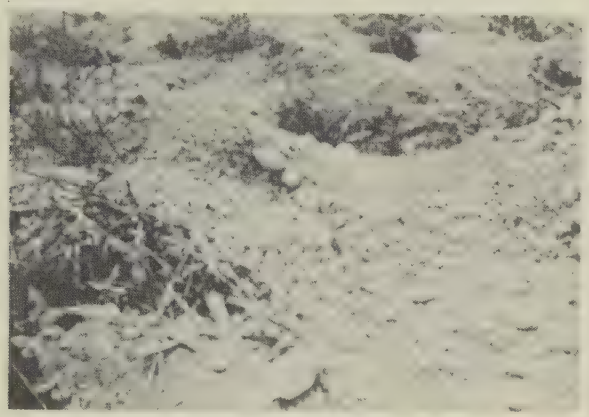

FIG. 10. Eluvial deposit, northwest of Sete Lagôas, Minas Gerais. Crystals are picked from the dirt which is broken down by picks and shovels. A carry-all removes the material to the dump.

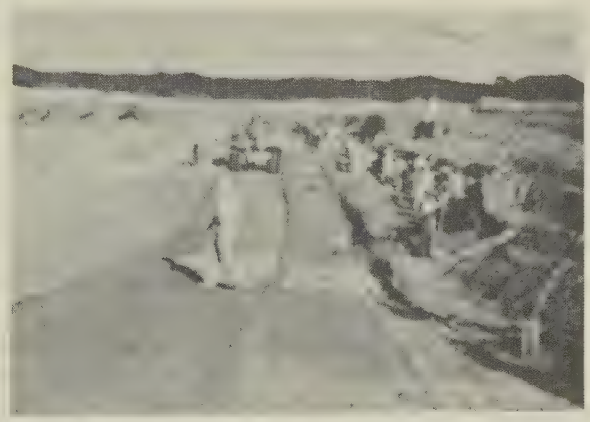

FIG. 11. Prospect pits in residual weathered rock representative of the hundreds of such pits near Sete Lagôas, Minas Gerais. The bedrock in this region is red shale. Rock fragments are bull quartz.

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[15] Knouse, F. L., Cristal de Rocha no Estado do Espirito Santo: *Div. fomento produção mineral* (Brazil), Avulso No. 55 (1944).

The Gameleira mine furnishes an excellent example of an alluvial de-deposit. It lies about 70 kilometers north of Curvelo, Minas Gerais. A bed of crystal-bearing yellow clay with maximum thickness of one meter underlies a varying quantity of red clay along the flanks of a very gently sloping hill. The thickness of the red clay overburden ranges from two to six meters.

SUMMARY OF MINERALOGY OF BRAZILIAN DEPOSITS

The mineralogy of the crystal deposits in bedrock is extremely simple. Quartz crystals are not produced in quantity from the same veins which produce other minerals. A black manganese oxide mineral frequently occurs in the crystal-bearing sections of the quartz veins and chlorite and limonitic stain are common. Several other minerals are reported, such as tourmaline, anatase, rutile, pyrite, an amphibole and clay minerals. A crystal supposed to have come from one of the mines in quartzite in Baia contains a small fairly perfect diamond included in the cap. Inclusions of pyrite are very rare. Quartz said to have originated near Xique-Xique, Baia, is yellow in color and contains fluid inclusions, hydrogen sulphide being given off when scratched. Shearer[16] reports a minute quantity of associated pyrite and arsenopyrite from deposits near Pium, Goiaz. Chlorite is commonly included in the crystals from the Pedra Verde mine in the Mimoso district of northern Baia. More thorough mineralogical study will undoubtedly add to the list of associated minerals. These associated minerals are, however, rare except for the oxides of manganese and iron, clay minerals and chlorite, so rare that specimens of them from crystal mines are unusual.

The crystals occur in vugs or pockets in the milky vein quartz. The ratio of milky or cloudy bull quartz to clear crystal varies but has been estimated as lying between 10,000 to 1 and 1000 to 1.

Quartz crystals occur in the veins in all sizes from the most minute to those famous crystals which weigh over five tons each. Some idea of the size distribution may be had from the following figures calculated from records of the weight classification of material purchased by the U. S. Government over a part of the war period.

The statistics show the greater rarity of the heavier (larger) crystals but also indicate that a surprising percentage fall in the 300–700 gram (approximately ½ lb.–1½ lbs.) category. It should be pointed out that there are certain limitations on the data since the figures were not compiled for the purpose to which they are being put in this paper although they nevertheless serve as a basis for a quantitative estimate. The figures represent size distribution of crystals in the 200–10,000 gram weight class which have been prepared for market and sold to the U. S. Government. Actually one end of many of the crystals may have been trimmed to remove cloudy areas; therefore, the figures indicate smaller-sized crystals than would actually be the case if the data related to newly mined untrimmed material. To an important degree material under 200 grams also finds a market and is very plentiful. It may be estimated that there are at least twice as many crystals of 100–200 gram weight class as all the larger sizes.

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[16] Shearer, H. K., Crystal Producing Areas of Northern Goiaz and Southeastern Baia, Brazil: Unpublished FEA Report, 1943.

QUARTZ CRYSTALS PURCHASED FOR NATIONAL STOCKPILE DURING PART OF THE WAR PERIOD*

Crystal Weight Group Weight in Grams	*Percentage of the Total Number of Crystals Which Were in Each Weight Group*
200– 300	55.5
300– 500	29.5
500– 700	10.4
700– 1,000	2.1
1,000– 2,000	1.8
2,000– 3,000	0.5
3,000– 4,000	0.2
4,000– 5,000	<0.1
5,000– 7,000	<0.1
7,000–10,000	<0.1

* Data based on over 1½ million crystals.

It is noteworthy that little of the clear quartz on the market is without a recognizable crystal face. Many of those unfaced crystals sold are fragments of larger crystals which did occur with natural faces present. From figures available it appears that the percentage of material in the 200 gram to 10,000 gram weight range which contains no recognizable face is from 5 to 20% of the total. Examination of the mines also points to the same conclusion. Anhedral quartz in the veins is almost entirely milky; translucent patches are rare or absent. While this might be expected, this contrasts with the condition of the massive quartz in some of the North Carolina-Virginia quartz veins in which translucent but not clear areas of vein quartz are sometimes seen.

GEOLOGY AND MINERALOGY IN RELATION TO COMMERCIAL PRACTICE AND SUPPLY

The interdependence of the mineralogical and geological factors, commercial practice and the problem of supply is well illustrated by the wartime crystal program; mineralogical differences in quartz crystals are reflected in crystal classification and affect commercial practice. Crystals are classified by weight in the Brazilian market. Twelve weight groups are recognized: less-than-200 grams, 200–300 grams, 300–500 grams, 500–700 grams, 700–1000 grams, 1–2 kg., 2–3 kg., 3–4 kg., 4–5 kg., 5–7 kg., 7–10 kg., and 10 kg. up. Crystal is further subdivided according to quality grades A, B, C, and D. Both the quality and weight classes have acquired legal status due to the incorporation of these in the Brazilian *tabela*, a table of prices used in levying export taxes.

The procedures followed in the manufacture of oscillator plates are adapted to the shape and size of the crystals utilized, but it is generally not practical to use shape as a basis for classification. The National Bureau of Standards has used the shape of quartz crystals in classification of the material in the less-than-200 gram weight group. A minimum length parallel to the *c*-axis and a minimum diameter perpendicular to the *c*-axis is required. Shape is not considered in classifying the stones in the heavier weight groups.

The shape of the commercial crystal is affected by the practice of trimming stones to remove the poor quality material. It is particularly common for quartz crystals to contain veils toward the base of the crystal and these are removed by expert trimmers who take advantage of the fact that crystals break easily along planes roughly parallel to rhombohedral faces.

Several features of the geology of the crystal deposits in Brazil which have been described have an important bearing on quartz crystal supply. The deposits are individually very small producers and are distributed over a large area, localized in four belts which do not coincide with the most populated regions of the country. As has been pointed out most of the mines produce less than 500 pounds of radio quality crystal per month and many only 50 or less, so that the total Brazilian production comes from several hundred deposits. The sedimentary rocks in which the deposits occur are frequently deeply weathered, allowing open pit mining without the use of explosives or machinery. Thus great numbers of individuals are engaged in independent mining in their own diggings. These people lead a nomadic existence, moving from one area to another because most small deposits are soon worked out and more favorable ground is opened elsewhere. In most areas transportation is difficult and so a condition of local trading has sprung up where the miner sells the crystal to a local merchant rather than direct to the quartz exporter. The merchant accumulates crystal and then disposes of it to another buyer. The crystal usually passes through many hands before reaching the exporter. This results in an appreciable time lag between production and the arrival of the quartz in the ports of Rio and São Salvador from which it is exported (Fig. 12). The various size and quality classifications used by the exporter further complicate the supply situation, particularly since so many producers are involved. The grade of the material and the judgment of quality are the considerations which determine the price to be paid or received, but because of the difficulty of appraisal much crystal is traded in lots at an overall price.

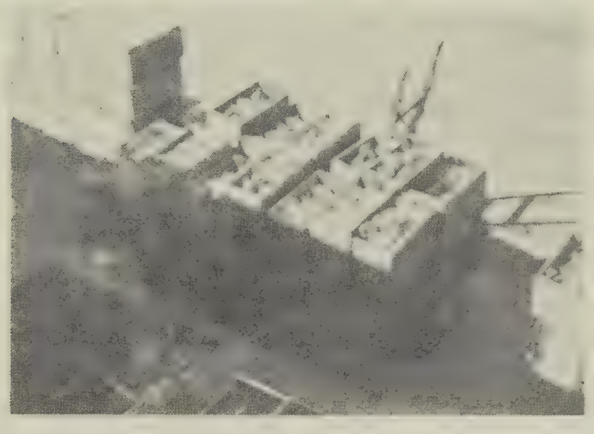

FIG. 12. Quartz boxed for boat shipment to U. S. Graded by weight class and quality. Rio de Janeiro.

Price per pound paid for crystal in the United States varies depending on the weight of the stone, and the percentage usability as indicated by the grading scheme. As an indication of the wide range of price it is sufficient to note that quartz from the government stockpile is sold at prices varying from $2.25 to $46.20 per pound.

SUMMARY

Quartz crystal production is almost entirely confined to Brazil. The price structure, the classification terminology and the methods of classification are all derived from Brazilian experience. Commercial practices are related to geologic features of the quartz crystal deposits.

The deposits of quartz crystal in Brazil appear to form a "mineral province." Features which characterize this province that appear to be significant may be summarized as:

1. The deposits lie in four elongated belts, widely separated and roughly parallel to one another.
2. Most of the deposits in bedrock are in Paleozoic sedimentary rocks which are not highly folded.
3. Most of the area in which the deposits occur is characterized by deep weathering.
4. Most of the deposits in bedrock are quartz veins, or flat-lying blanket deposits of milky quartz roughly parallel to the enclosing sediments, or a combination of these two types. The clear crystal usually occurs in vugs or pockets and appears to have formed later than the bulk of the milky quartz.
5. The deposits are characterized by very few minerals in important quantity other than milky quartz.
6. Veins are apparently unrelated to other mineralization.
7. The veins are exploited for no product other than quartz crystal.
8. With few exceptions single deposits produce not more than 500 pounds of usable crystal per month.

Information available indicates that quartz deposits in bedrock throughout the world, except those in Australia, conform to most of the characteristics listed above. They depart from the Brazilian type in the nature of the surrounding rocks and the extent of the weathering. The Australian deposits differ in many respects. The recurring habit noted in some localities in Brazil and in Australia of the crystal occurring in subsidiary veinlets or branching pods, offshoots from the main body, is perhaps worthy of note.

In summary, most of the characteristics listed as typical of the Brazilian province appear from present data to be generally applicable to quartz crystal deposits.

> Crystal is subject to numerous defects, sometimes presenting a rough, solder-like, substance, or else clouded by spots upon it; while occasionally it contains some hidden humour within, or is traversed by hard and brittle inclusions. . . . Some crystal, too, has a red rust upon it, while, in other instances, it contains filaments that look like flaws. . . . *Pliny*, XXXVII, chap. 11.

THE INSPECTION AND GRADING OF QUARTZ

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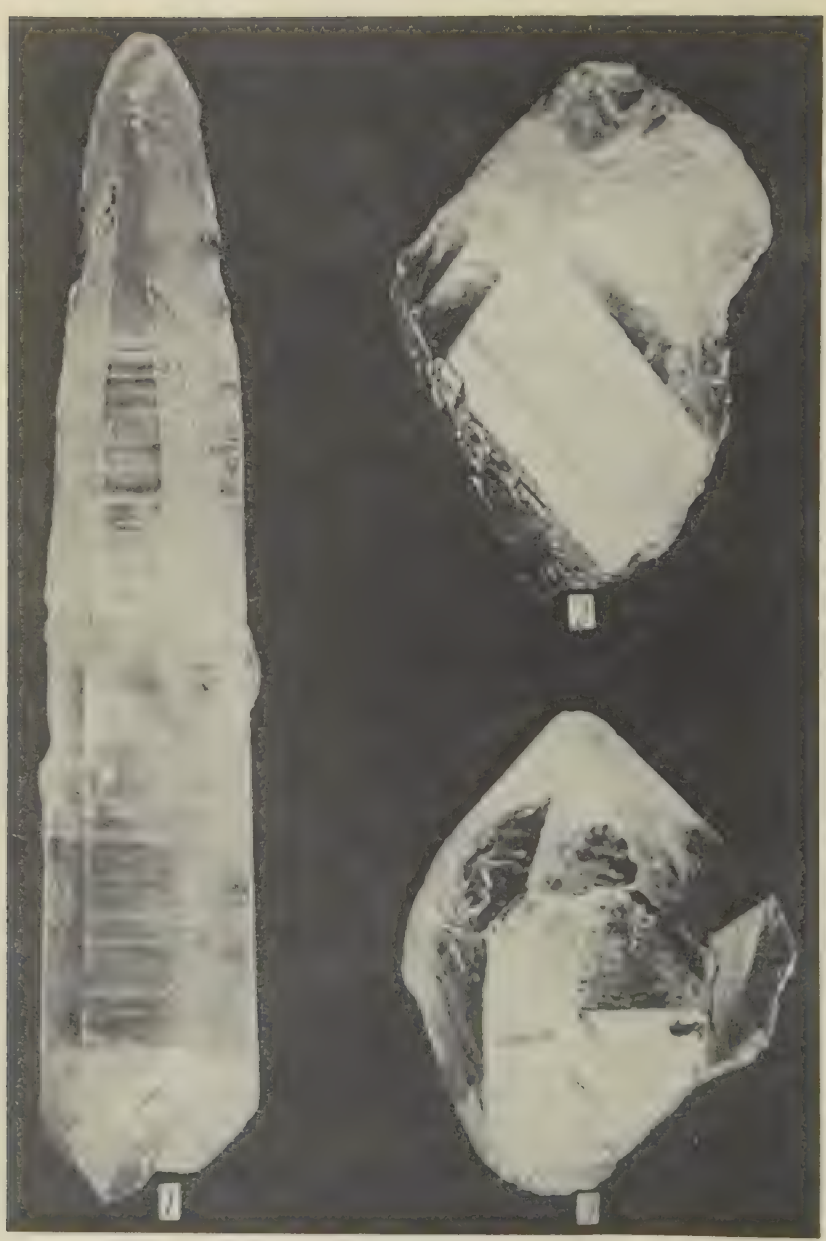
ABSTRACT

To be usable for radio frequency oscillators, a quartz crystal must have part of its volume free of twinning and detrimental inclusions. Dauphiné ("electrical") twinning may sometimes be detected upon the surface of the crystal, or by etching crystals or sections. Brazil ("optical") twinning, intergrowths of right and left quartz, is easily detected by viewing a crystal, immersed in oil, and between crossed polaroids, in the direction of the optic axis. An oil bath, so set up, has been called a quartz inspectoscope. It also permits the quartz to be scanned in an intense beam of light for cracks, bubbles, inclusions, and the inexplicable oriented linear clouds in V-shaped clusters called "blue needles." The apparatus is not only useful in grading quartz for "usability," but for inspection of sections, bars, wafers, and blanks to avoid useless processing. A portable field model, using sunlight, is described. An example of the rare type of twinning according to the "combined laws" is illustrated.

INTRODUCTION

The usefulness of quartz for radio-frequency oscillators depends upon the absence of deleterious matter and structures that affect its elasticity or cause electrical anomalies.



FIGS. 1–3. Typical Brazilian crystals of radio grade. Fig. 1: a "candle"; Fig. 2: a crystal with a large *s* $(11\bar{2}1)$ plane, showing characteristic striations parallel to edge *s*:*r*; Fig. 3: a Dauphiné ("electrical") twin, showing the characteristic sutured boundary on *m* (1010).

We are not particularly concerned with the obvious inclusions of rutile, tourmaline, hematite, pyrite, mica, chlorite, or moving bubbles that are of interest to mineralogists but with quite common defects that occur in *eye-clear* rock crystal. Twinning is as prevalent in quartz as it is in plagioclase feldspar, and quartz suitable for cutting flawless crystal balls may consist of polysynthetic twinned laminae that destroy its usefulness for radio purposes. Limpid quartz is apt to be filled with invisible bubbles, or the remarkable linear clouds in V-shaped clusters called "blue needles."

Quartz oscillators may be cut from either left or right quartz, but the presence of both in the same plate results in electrical anomalies. Since quartz oscillators are cut in a definite crystallographic orientation, a plate oriented with respect to one part of a twin will be misoriented for the other.

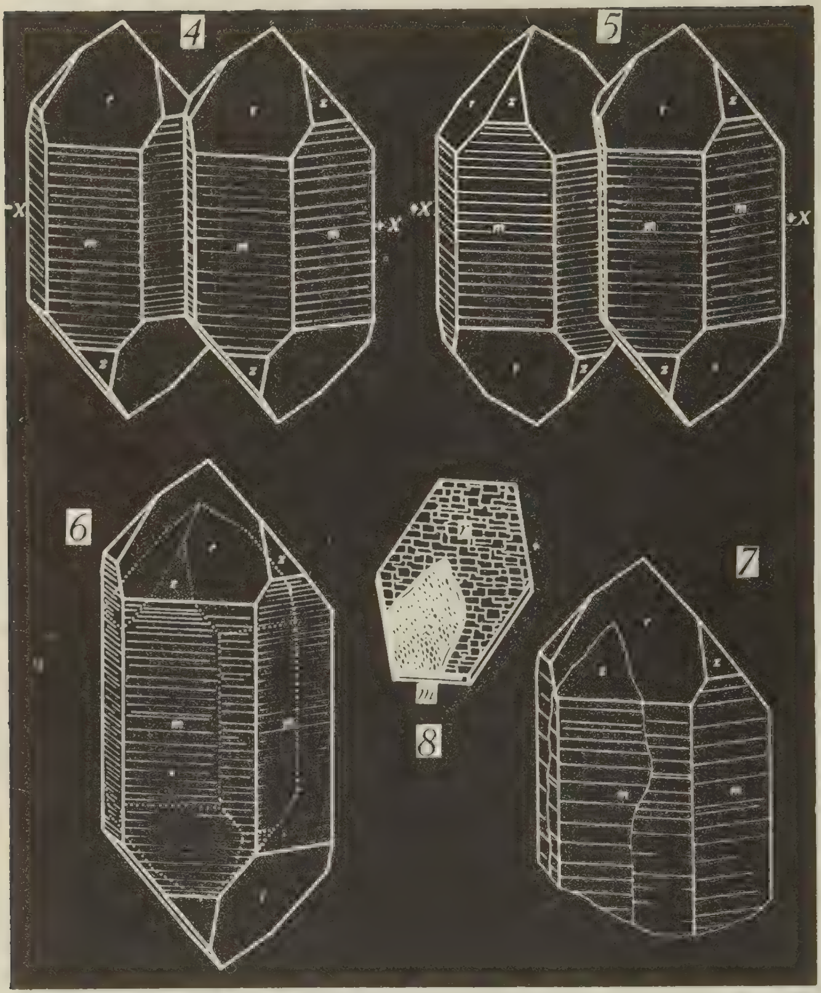
The presence of some minute bubbles and "blue needles" may be tolerated in large plates vibrating at low frequency (such as X-cuts and filter plates), but it is obvious that a bubble in a small plate will occupy an appreciable volume of the whole plate and may seriously affect its elastic properties. It is now the general practice to etch oscillator plates to their nominal frequency-thickness; bubbles and "blue needles" are rapidly attacked by the etching solutions, and their absence should be assured.

It is not only necessary to grade the quartz on purchase, but to inspect it at various stages of manufacture to avoid useless processing. It is also possible to plan the cutting operations to get a maximum yield from twinned quartz, and rules to accomplish this will be given in an accompanying paper.

Twinning in Quartz

Untwinned quartz is quite rare, and it is unusual to find a crystal which is not twinned according to both the Dauphiné and Brazil laws.

Dauphiné ("electrical") twinning is the designation of Dauphiné twins by radio engineers: an intergrowth of either two right *or* two left individuals, at 180° to each other, with c as the twin axis (Figs. 4–8). The rhombohedral faces r $(10\bar{1}1)$ and z $(01\bar{1}1)$ become coplanar, as do also m $(10\bar{1}0)$ and $\bar{m}$ $(\bar{1}010)$. Such twins are often recognized by the sutured boundary on m $(10\bar{1}0)$, or by a difference in the sheen of the coplanar forms r and z on the apex faces (Fig. 9). Dauphiné twins are also recognizable by the presence of s $(11\bar{2}1)$, and x $(51\bar{6}1)$ on both edges of a prism face, when the normal, untwinned trigonal symmetry calls for these faces only on alternating prism edges. This type of twinning is



FIGS. 4–8. Parallel growth and Dauphiné ("electrical") twinning. Fig. 4: a parallel growth; Fig. 5: a Dauphiné twin: an intergrowth of two right *or* two left crystals with the polarity of their X-axes reversed; Fig. 6: an idealized intergrowth of the individuals of a Dauphiné twin showing *r* and *z* as coplanar; Fig. 7: a more natural representation; Fig. 8: appearance of an etched wafer showing the characteristic "ripple" and "shingle" structure developed by coplanar *z* and *r*, respectively.

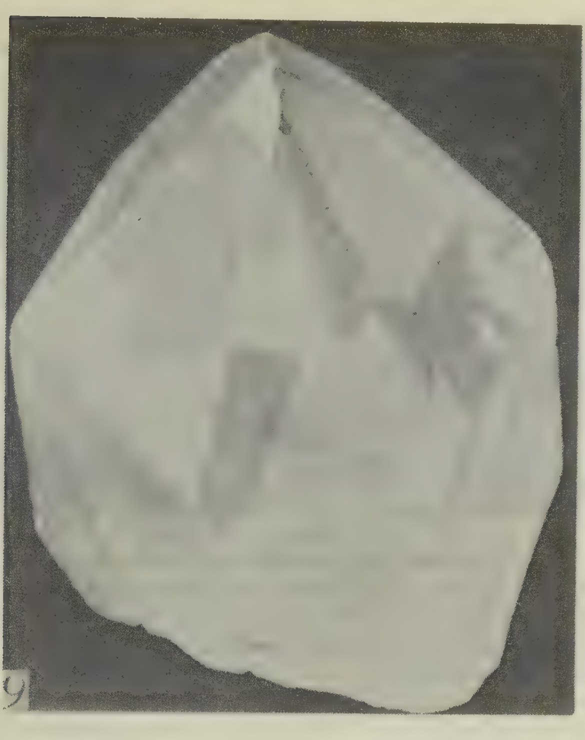


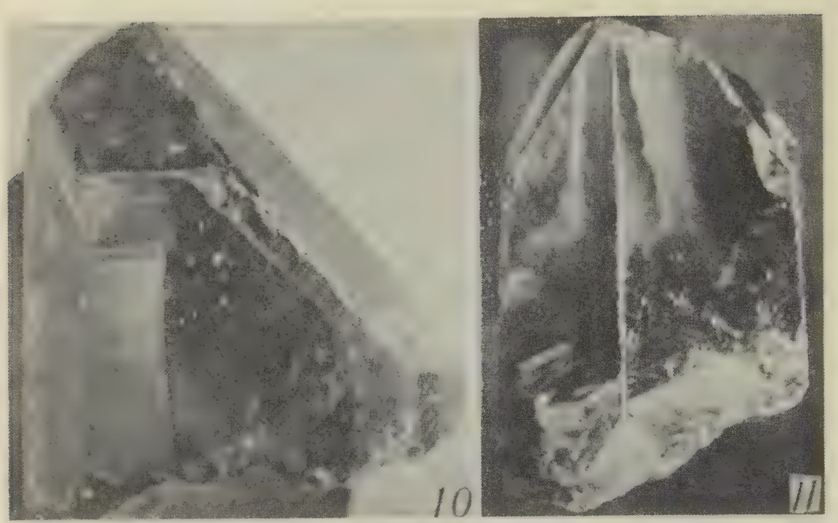
FIG. 9. In Dauphiné ("electrical") twinning, r $(10\bar{1}1)$ and z $(01\bar{1}1)$ are coplanar, but often distinguishable by differences in sheen, due to natural etching. (Photograph through the courtesy of Lieut. William B. Gray, Jr.)

called electrical twinning because it results in a reversal of the polarity of the electrical axis ($X\ [11\bar{2}0]$) at the twin boundary.

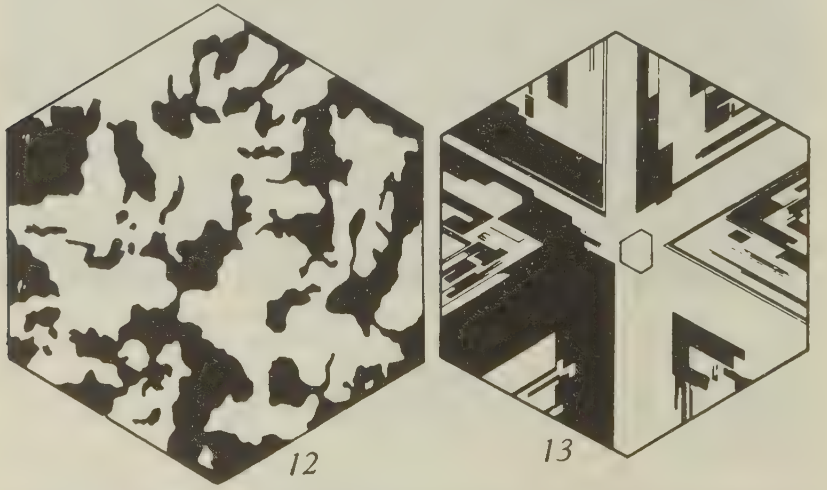
In marked contrast to twins of the Brazil law ("optical twinning") the twin boundaries of Dauphiné twins are quite irregular, and this serves to distinguish the two in etched sections (Figs. 12–13).[1] In Dauphiné, "electrical" twins, the coplanar areas, r and z can be distinguished in etched wafers by their difference in translucency and structure; r developes a characteristic "shingle" structure, while z develops a less symmetrical "ripple" pattern (Fig. 8); this is further discussed in the following paper.

---

[1] Leydolt, Franz, Über eine neue Methode, die Struktur und Zusammensetzung der Krystalle zu untersuchen, mit besonderer Berücksichtigung der Varietäten des rhomboedrischen Quarzes. *Sitzber. d. kais. Akad. d. Wiss. Wien* (Math.-Natur. Classe) XV (1) 59–81, **1855.**



Figs. 10–11. Two views of a Dauphiné ("electrical") twinned crystal, showing a sutured boundary on the prism *m* $(10\bar{1}0)$, and the equal development of two apex faces and zonal development of these with *m* $(10\bar{1}0)$. The latter, resulting in a pseudo-prismatic appearance of the apex faces with long edges in contact with *m* is diagnostic in detecting electrical twinning. (Photographs by NAP.)



Figs. 12–13. Etched sections reveal twinning strikingly, and Dauphiné ("electrical") twinning can be distinguished from Brazil ("optical") twinning by the irregular boundaries developed in the former (Fig. 12) in contrast with the straight boundaries developed in optical twinning (Fig. 13). These sections are perpendicular to the optic axis. (After Leydolt.[1])

A Dauphiné twin may consist of a pair with an irregular boundary between them. Other crystals contain many quite irregular patches—which suggests that the twinning may be secondary; that is, due to stresses set up through sudden changes of temperature and pressure. A small plate heated in an oven (not necessarily to the inversion point) will twin on sudden cooling, and etching will reveal curved boundaries symmetrical to the edges of the plate.

Brazil "optical twinning" is the name applied to twinning after the Brazil law, because it is readily detected by examination in polarized light. This kind of twinning is an intergrowth of the two enantiomorphous forms: of right and left individuals.[2]

The story of the discovery of this kind of twinning is recorded by Sosman:[3]

> "Twinning in quartz was first placed on record by Weiss[4] in 1816. He described and figured clearly the orientational (Dauphiné) type of quartz twins. The chiral (Brazil) type he appears also to have observed, though rarely, but spoke of it cautiously as a kind of intergrowth which might possibly be explained as a case of twinning allied to the orientational. The difficulty was simply that the difference between the two alternative choices, —positive versus negative forms on the one hand, and right versus left on the other,—had not been made clear.
>
> "Herschel[5] happened upon some twinned crystals in the course of his pioneer work on the correlation of optical activity and face development in quartz and was puzzled to find one specimen which had trapezohedral faces perfectly distinct and in contact but 'tending opposite ways around the summit.' Unfortunately it was 'in the possession of Mr. Brooke,' and Mr. Brooke apparently valued it too highly to let it be damaged in the interest of science, so Herschel was not allowed to section it and test its rotatory power for polarized light. The sympathy of every true mineral collector will be with Mr. Brooke, even though it did postpone an important scientific discovery for twenty-five years.
>
> "The explanation for this peculiar crystal was not forthcoming until G. Rose[6] published his comprehensive work in 1846. He was the first to show that the apparently homogeneous crystal with the opposite tending faces is really a compound crystal, twinned according to the Brazil law."

---

[2] Lord Kelvin (Baltimore lectures, 1904, p. 640) called it *chiral* twinning (from the Greek, for hand). The optical rotary power due to handedness has been called *chirality.*

[3] Sosman, Robert B., *The Properties of Silica*, p. 196–197, Chemical Catalog Co., New York (1927).

[4] Weiss, C. S., Über den eigenthümlichen Gang des Krystallisations-systemes beim Quarz, und über eine an ihm neu beobachtete Zwillingskrystallisation: *Ges. Nat. Freunde Mag. Berlin* **7**, 163–181, pl. 4 (1816).

[5] Herschel, J. F. W., On the rotation impressed by plates of rock crystal on the plane of polarization of the rays of light, as connected with certain peculiarities in its crystallization: *Trans. Cambridge Phil. Soc.* **1**, 43–52 (1821).

[6] Rose, Gustav, Über das Krystallisationssystem des Quarzes: *Physik. Abh. Köngl. Akad. Wiss. Berlin,* 1844: 217–274, pl. 1–6 (1846).

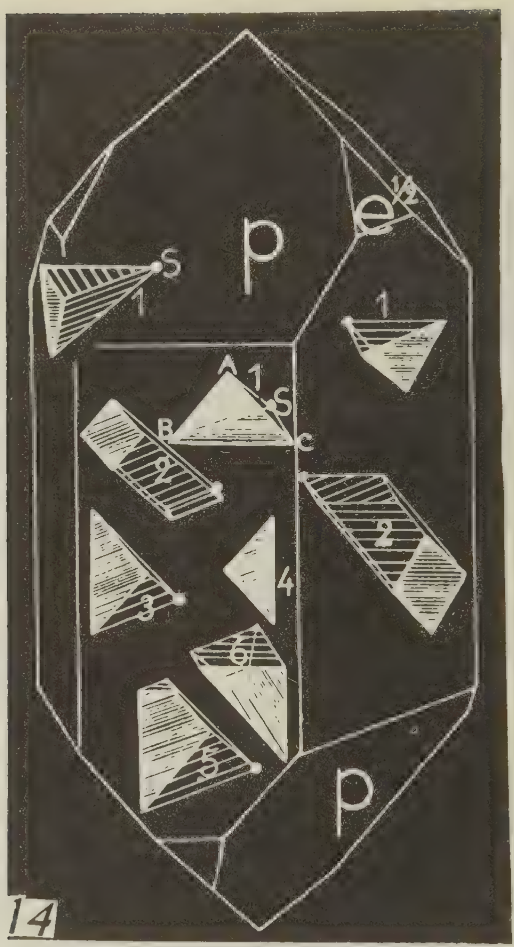


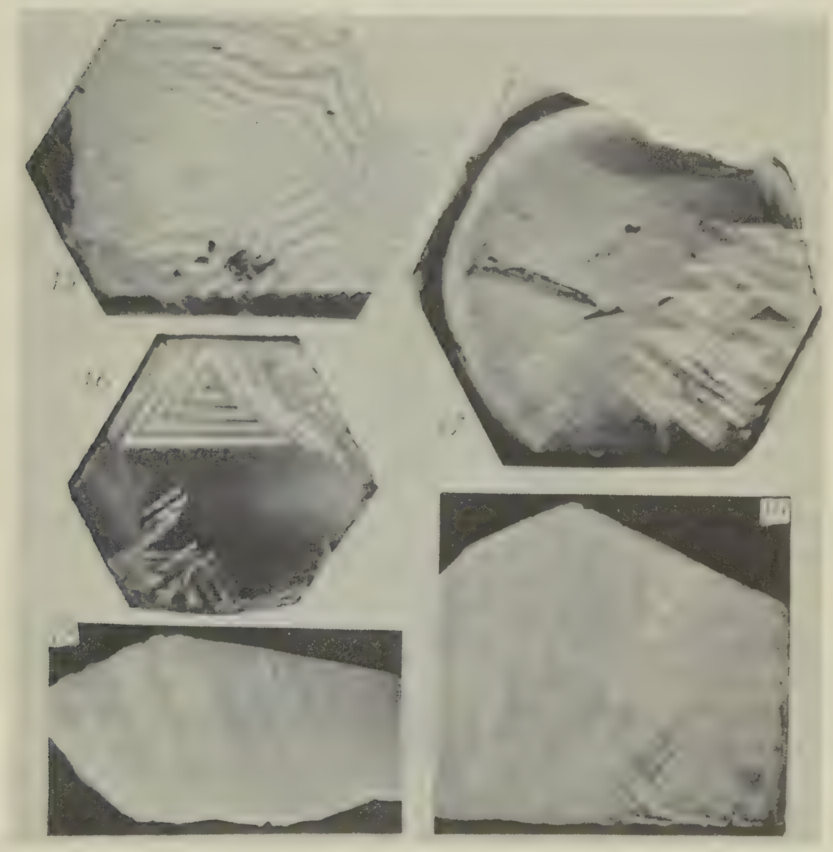
FIG. 14. Brazil ("optical") twinning is the intergrowth of right and left quartz. It consists of laminae of one hand enclosed within a host crystal of the opposite hand. The remarkable shapes assumed by these "satellites" are shown in the idealized perspective of the Abbe C. Gaudefroy,[7] who listed as the principal forms $m(e^2)$ $10\bar{1}0$, $r(p)$ $10\bar{1}1$, $z(e^{1/2})$ $01\bar{1}1$, s $11\bar{2}1$, and $c(a^1)$ 0001.

[7] Gaudefroy, C., Sur les groupements de cristaux de quartz à axes parallèles: *Bull. soc. franç. min.* **56,** 5–63 (1933).

Lamellar twinning was discussed by John W. Judd: Additional note on the lamellar structure of quartz crystals, and the methods by which it is developed: *Min. Mag.* **10,** 123–135 (1892).

Similar twinning in cinnabar was described by Waldemar Lindgren: *U. S. Geol. Surv. Bull.* **61** (1890).

Crystals twinned according to the Brazil law and resembling the idealized drawing of Rose, which has been copied in all text-books, are rare, and their existence requires confirmation. This type of twinning is seen rather as a growth of laminae of one hand within a host crystal of the opposite hand (Figs. 14–19). The shapes assumed by these inclusions are remarkable: they suggest crystals of the pedial class of the triclinic system, in that they are triangular polygons, and often with but one form



FIGS. 15–19. Brazil ("optical") twinning may be detected by viewing crystals (immersed in oil between crossed polaroids) in the direction of the optic axis. The contour of the base or termination is seen in bands of color expressing various degrees of rotation of the plane of polarization because of varying thickness of quartz (Fig. 15). Laminae of the opposite hand are seen as triangular areas (Figs. 16–17). Etching of sections localizes the twinning (Figs. 18–19). (Photographs 15, 18–19 by NAP.)

of a kind. Not only are they of a habit entirely different from that assumed by the host crystal, but they may show a well developed basal plane! The form of these "satellites" was described by the Abbé C. Gaudefroy[7] in 1933, then president of the Sociéte Française de Mineralogie, and the principal types are reproduced in his idealized perspective (Fig. 14). As a rule the laminae are quite thin, and many laminations of apparently the same dimensions may occur rhythmically spaced above one another in the host, and invariably parallel to the major rhombohedral faces (r $10\bar{1}1$) (Fig. 17).

Most quartz crystals are intimate intergrowths of polysynthetic laminae of right and left quartz, and it is principally in Brazil that quartz crystals occur relatively free of the twinning to which that country has given its name! Quartz of radio quality from Brazil usually shows the parasitic laminae at the margins of the crystals, and the interior may be quite free of them.

Twinning According to the Combined Laws

Electrical twins (Dauphiné law) are described as two right, *or* two left individuals, at 180° to each other with respect to the theoretical twin plane $11\bar{2}0$; the faces r ($10\bar{1}1$) and z ($01\bar{1}1$) are coplanar. Optical twins (Brazil law) are simply intergrowths of the two left and right enantiomorphous form: the forms r, z, m on one individual are parallel to r, z, and m of the other.

An example of the combined laws[8] is shown in Fig. 20. Two parallel planes perpendicular to the electrical axis (a, X-axis) have been cut on the crystal; one was etched and the other polished: the photograph shows the view looking through the polished (+) X-plane to the etched (−) X-plane at the back.

From the parallelogram light figures (described in the accompanying paper on Orientation), the two parts may be considered as an electrical twin of a right and a left crystal, or as an optical twin in which the right and left crystals are 180° to each other, with r and z coplanar.

It is characteristic of electrical twins to have irregular boundaries, while optical twins have straight boundaries: it will be noted that this specimen shows both; the boundaries parallel to r ($10\bar{1}1$) are plane, as it should be in an optical twin: the other boundary is quite irregular.

---

[8] Found by Mr. Isaac Herr of the Commercial Crystal Company of Lancaster, Pa. Other examples, discovered by Mr. Hoyt Brubaker, were generously given to the writer by Mr. P. R. Hoffman of Carlisle, Pa., for study. L. A. Thomas, *Nature*, **155**, 425 (April 7, 1945) suggests the name "compound optical twinning" for the combined laws.

DETECTION OF "OPTICAL" TWINNING IN POLARIZED LIGHT

Twinning according to the Brazil law is called "optical" twinning because it is so readily detected by viewing the quartz between crossed polaroids in the direction of the optic axis. This may be readily demonstrated by immersing a *clear* quartz crystal in a beaker or culture dish filled with an oil of proper index. The beaker is set upon a pair of glass plates between which is a film of polaroid (a diffusing ground glass plate is also desirable). The plates are placed upon a laboratory tripod—an electric light within the tripod may be used for illumination. A second polaroid, crossed to the first, is set upon the beaker. Upon viewing the quartz crystal at a slight angle to the optic axis, the twinned laminae will be seen as thin, spectacularly colored plates (Figs. 15–17). The phe-

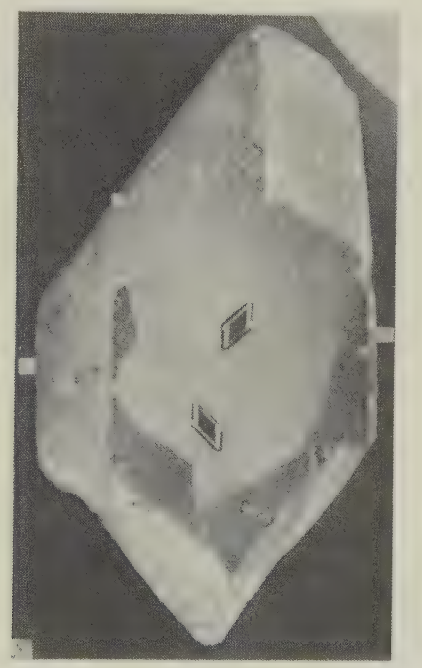
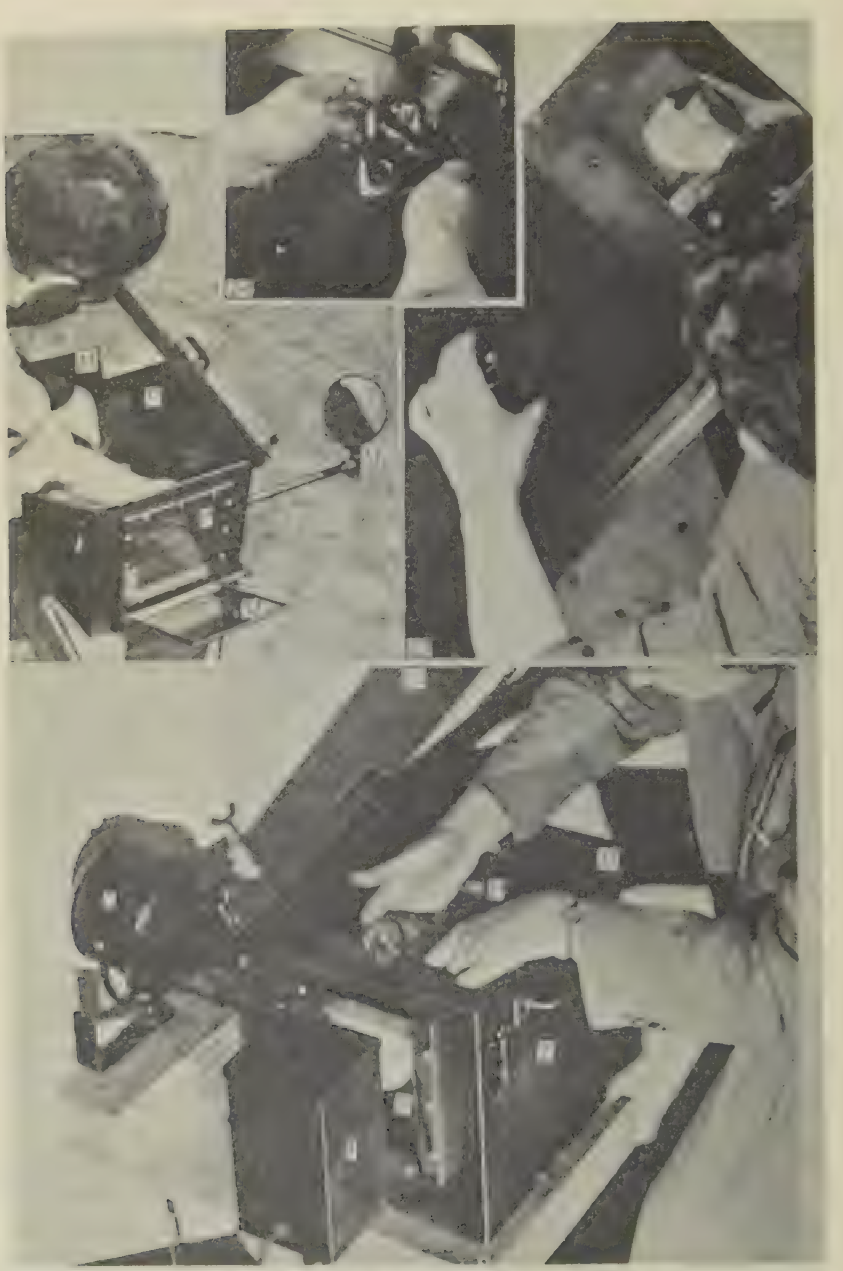


FIG. 20. A crystal twinned on the rare "Combined Laws." View perpendicular to electrical axis [11$\bar{2}$0]: through the polished face [$+X$] to etched face in back [$-X$]. Sketched in are the light figures seen on the etched surface on viewing a point source of light. The central area has the character of both an optical twin (straight boundaries parallel to r (10$\bar{1}$1)), and of an electrical twin (the irregular boundary). About 2/5 natural size.



FIGS. 21–24. The quartz inspectoscope is simply an oil bath in which a quartz crystal can be viewed between crossed polaroids to detect optical twinning; it also permits scanning of the crystal in an intense beam of light for flaws and inclusions. Fig. 21: the Klieg model used in the plant: the tank (T) has a pair of crossed polaroids (P), a light source (I) and a view-

nomena is striking only if viewed within a few degrees of the optic axis, and it is essential that the quartz be immersed in an oil approximating the indices of refraction of quartz .The most satisfactory liquid is ortho-free tricresyl phosphate ($n=1.55$) sold under the trade name of *Lindol.*[9]

In general, the laminae have the appearance of right triangles: with the base of the triangle parallel to the prism face with which it is usually in contact; the hypotenuse parallel to a prism face at 120° to the first (parallel to an $m:r$ edge, or an X axis); and the third side parallel to an $r:r$ edge (Y axis; prism normal). Other plates appear as equilateral triangles, with all three sides parallel to traces of prism faces ($m:r$ edges).

The usefulness of this method depends upon the fact that the parasitic laminae are thin and wedge-like, and they rotate the plane of polarization in the opposite direction to that of the host. The polysynthetic twin laminae are thin enough to show several orders of interference colors, while the host is so thick that interference colors are seen only as colored bands fringing the base of the crystal and expressing variations in thickness of the contour of the latter (Fig. 15). The interference colors shown by various thicknesses of quartz are tabulated in Table 1.[10]

THE QUARTZ "INSPECTOSCOPE"

The quartz inspection apparatus used in crystal-cutting plants is essentially a tank in which the quartz can be immersed in oil and examined in polarized light, as well as in the intense beam of a spot-light (Figs. 21–24). Often it was merely a fish aquarium, the edges of which had been

[9] Obtainable from the Celanese-Celluloid Corporation, 180 Madison Avenue, New York City, for about $0.285 per pound in 5 gallon lots. Specify *ortho-free*, a grade which seems to be free of dermatological hazards. After immersion, crystals are readily cleaned with laundry soap and water.

[10] More than a century ago, Fresnel showed that quartz was birefringent in the direction of the optic axis. Sosman quotes for ω and ϵ, respectively 1.5441887 and 1.5442605, or a birefringence of 0.0000718. Plane polarized light traveling in the direction of the optic axis is separated into two circularly polarized rays, of opposite sign and different speeds, which emerge to form plane polarized rays with the interference color composed of the resultants of the rays which were not extinguished. Airy showed that rays not parallel to the optic axis were transmitted as two sets of elliptically polarized waves. G. Szivessy (*Fort. Min. Krist. Petr.* **21**, 111–168 (1937)) has shown that α quartz is optically inactive at 56°10′ to the optic axis. Rays traveling perpendicular to the optic axis are plane polarized. Full discussions of the rotatory power of quartz will be found in the works of Sosman and Tutton.

---

ing mirror (M); (L) is an intense spot-light. In the field, a circular mirror is used to reflect the direct rays of the sun for scanning the crystal for flaws and inclusions (Fig. 22), while another mirror (M) is used to reflect light from the sky overhead in searching for optical twinning. Fig. 23: the crystal is being viewed in the mirror (along the optic axis direction) for twinning. Fig. 24: scanning the crystal in an intense beam of light for inclusions. (Photographs, courtesy of NAP.)

sealed with asphalt paint, and which had been fitted with screens of polaroid at the ends, similarly sealed against seepage of oil. Illumination was provided by a pair of 40 watt electric light bulbs, diffused by ground glass or opal glass at one end, and a mirror was provided for viewing the quartz at the other end.

TABLE 1. ROTATION OF THE PLANE OF POLARIZATION OF QUARTZ*

Color extinguished	Fraunhofer line	Wave length λ in $\mu\mu$	Rotation per mm. at 20°C.	Thickness necessary to rotate 90°	Thickness necessary to rotate 180°	Interference color between crossed nicols
Red	A	760.4	12.65°	7.15	14.30	Green
	a	718.36	14.30	6.29	12.58	Blue
	B	686.71	15.75	5.71	11.42	
Orange	C	656.21	17.31	5.19	10.39	
Yellow	D_2	589.513	21.69	4.14	8.29	Violet
	D_1	588.912	21.725	4.11	8.23	
Green	E	526.913	27.54	3.26	6.53	Red
Blue	F	486.074	32.76	2.75	5.49	Orange
Indigo	G	430.725	42.59	2.10	4.20	Brownish yellow
Violet	h	410.12	47.49	1.89	3.79	
	H	396.81	51.19	1.75	3.51	Yellow

* Johannsen, A., *Manual of Petrographic Methods*, p. 109. McGraw-Hill, New York (1914).

Such equipment was modeled after the quartz "inspectoscope" developed by the Bell Laboratories for the Western Electric Company, where mercury vapor lamps were used for illumination, and the light was filtered through a series of Corning color filters including an Illusion pink, a yellow shade yellow, and a Light Shade blue green, as well as a heat filter. This instrument had advantages over direct viewing tanks used in the Bureau of Standards, where the lower polaroid plate was in the vulnerable position of being at the bottom of the tank and was soon obscured by dirt or cracked by quartz crystals slipping from the operator's hand: moreover, the mirror image seemed to present a better picture of the twinning that could be gathered from scanning the crystal directly.

Field Model

A smaller, portable apparatus (Fig. 22)[11] was introduced for use in the field, but proved equally popular in crystal-cutting plants. It was designed for use in regions remote from electricity, and was illuminated by light reflected from the sky and the sun by a pair of folding mirrors: for that reason they became known in Brazil as "sun buckets." A tight fitting lid permitted transportation of the oil in the bucket.

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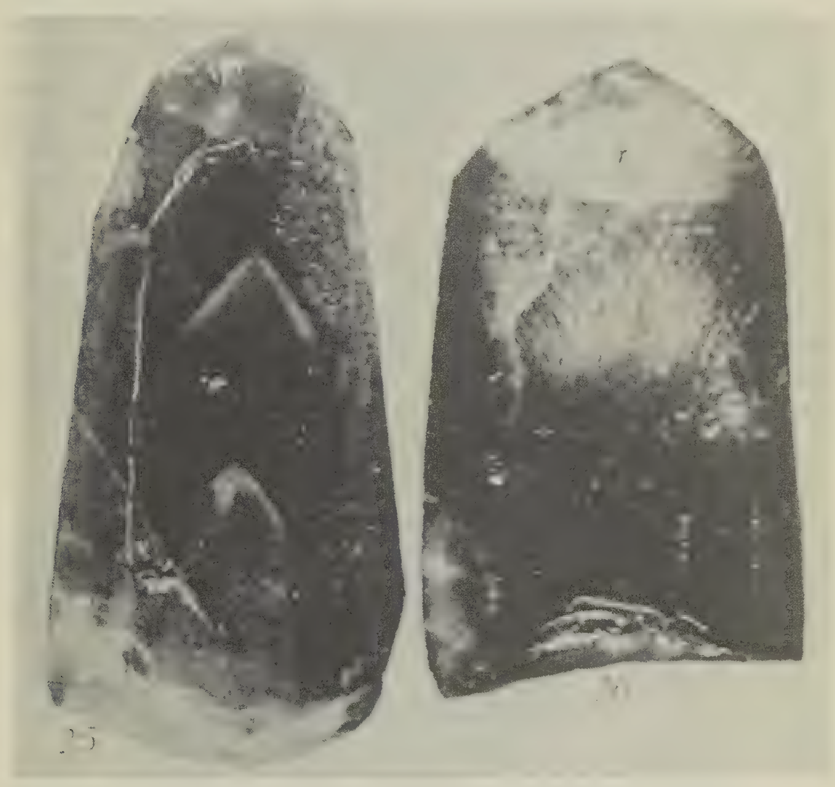
[11] Manufactured by Kliegl Brothers, 321 West 50th Street, New York City: a Klieg spotlight is provided for plant use.

Upon reaching a prospect, the tank is set upon a rock, with the sun to one side of the prospector (Fig. 22). The lid is unclasped and set up as a shield to exclude extraneous light. The mirrors are pulled out and turned to their reflecting angles. The light of the sky overhead or in front of the prospector is reflected into a polaroid window, and the mirror opposite it is adjusted for viewing quartz for optical twinning. Another, circular, mirror is set to reflect the sun's rays into the tank for examination for flaws, bubbles, and other defects: a metal shield hung over the tank can be slid over this window to exclude the direct beam during the examination in polarized light.



FIGS. 25–26. Eye-clear, limpid quartz is quite apt to show phantoms or "blue needles" on scanning the crystal, immersed in oil, in an intense beam of light.

Detecting Optical Twinning in Practice

The quartz must be thoroughly cleaned before inspection; moreover cloudy areas at the sides or base must be trimmed by means of a hammer, rock trimmer or saw: this is particularly true of such crystals as occur at Hot Springs, Arkansas in which the lower part is apt to be quite cloudy.

The clear crystal is held in the oil bath, with the optic (Z) axis direction (*c*), perpendicular to the crossed polaroids. The irregular base will be fringed by concentric color bands expressing variations in thickness of the margin of the crystal (Figs. 15–17). Twin laminae will appear as colored *triangular* blades along the side or shooting through the crystal, or cutting across the concentric color bands of the margin of the mass. By slightly tilting the crystal the position of the twinned areas can be approximately located and usable volume of the quartz, free of twinning, can be estimated.

Since it is necessary to orient the quartz precisely in order to see the twinning, some difficulty may be experienced at first with defaced quartz; the following rule will be helpful: Turn the mass horizontally in the oil bath until the color bands concentric with the margin appear or the quartz is at extinction; in the latter case, rotate it now about an axis parallel to one of the polaroid vibration directions.

FLAWS AND INCLUSIONS IN QUARTZ

An intense beam of light from a carbon-arc or a projector is used to detect flaws and inclusions in quartz. The quartz is scanned while it is submerged in an oil approximating it in index of refraction, and the effect is as revealing as the beam from a moving-picture projector through a dust-laden darkened room.

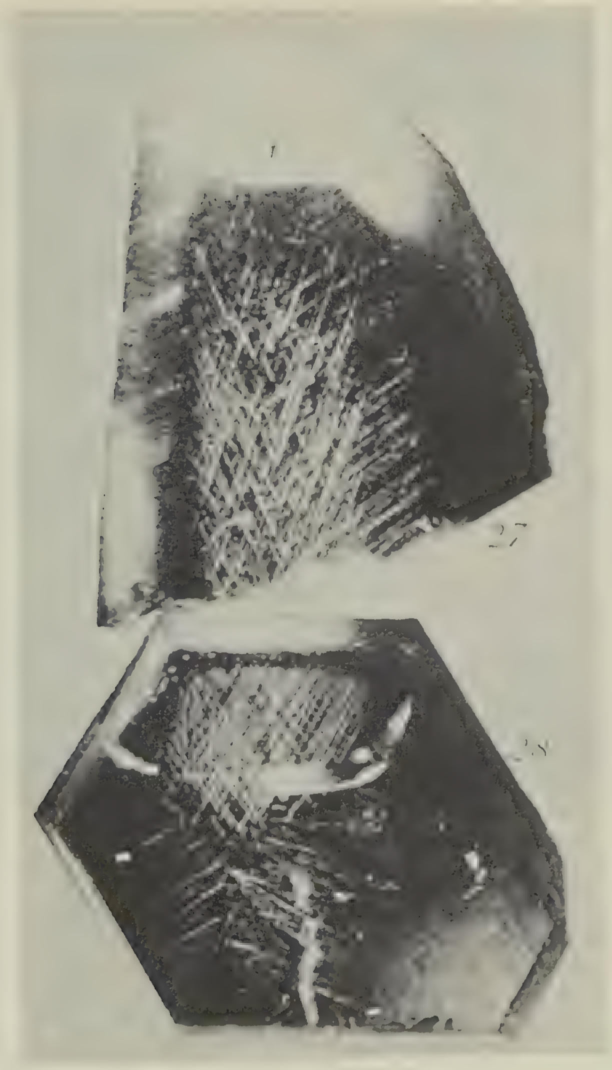
The principal defects are fractures, cavities, bubbles, "ghosts," and the remarkable "blue needles" and "blue feathers" (Figs. 26–28). Various designations used by quartz inspectors are:

(1) "Bubbles": bubble-like cavities, distinguished as "large" or "fine."

(2) "Veils," "heavy" or "fine": more or less continuous sheets of small bubble-like cavities.

(3) "Clouds" and "haze": aggregates of fine, bubble-like cavities.

(4) "Ghosts" are the familiar "phantoms" of mineral collectors (Fig. 25): earlier growths within the crystal, which become visible when a beam of light is reflected from the minute fractures or parting planes which outline them. They are also seen as alternating colorless and smoky zones within the crystal.



FIGS. 27–28. "Blue needles" or "blue feathers" are quite common inclusions of linear clouds in V-shaped clusters, with a preferred orientation of the limbs of the V parallel to an $r:z$ intersection edge, and the plane of the V parallel to an r face. The apex of the V always points to the base of the crystal, never to the terminated end. The reticulation is only apparent, the V's being at successive, often equidistant, levels. They seem to be systems of minute parting planes. (About 3/4 natural size.) The relation of the basal view (Fig. 28) to Fig. 27 is as follows: the edge which is common to the two is in contact, and the exact relation can be seen by folding Fig. 28 under Fig. 27 along this edge. (The prism face and basal section shown were kindly polished by William Van Horn.)

(5) "Blue needles" and "blue feathers" (Figs. 26–28): these are inexplicable linear clouds which appear bluish because of selective absorption of rays of others colors. They occur as pairs of needles forming a V, with the apex of the V *always* towards the base of the crystal, and the two arms of the V have a preferred orientation parallel to r:z edges; in other words, a V outlines a plane parallel to z towards the base of the crystal.[12]

The "blue needles" are not reticular aggregates, such as shown by rutile inclusions in rose quartz, although the photographs give this illusion through projection on the same plane of V's at successive, and often equally spaced levels below the surface. "Blue needles" are not uncommon in quartz, but patience and alertness are necessary in hunting for them, since they become visible only in certain orientations in a powerful beam of light while immersed in oil: for example, the basal view shown in Fig. 28 showed the blue needles only when the beam of light struck a prism face above a major rhombohedral face r $(10\bar{1}1)$. Under a magnification of 30x, using a binocular microscope, the V is seen to include a number of finer needles radiating from the same point but not in the same plane. These finer needles are seen to be very narrow planes with a cross fiber structure: a single needle may be the intersection of two such planes. The fibers seem to be parallel to parting planes. Jutting out from the needles, at almost regular intervals, and more or less at right angles to the needles, are flat spear-heads: these seem to have a central line, which may have been a fiber, and a structure which appears to be made up of triangles or rhombs. They diffract light into the circular dots seen on the needles as "dew-drops" in the oil baths. These structures are hard to resolve, even under a magnification of 150×, but they seem nothing more than systems of linear parting planes. This interpretation is supported by examinations of etched wafers, where the etching solutions have penetrated the blue needles: only linear cavities bounded by planes parallel to parting directions (r $10\bar{1}1$) are seen (Figs. 29–32).

The Portugese word "chuva" meaning a fine rain is said to be applied to "white needles" with "small bubbles" giving the appearance of dewdrops along a thin fiber.[13]

Tyndall effect is a bluish smoky appearance of a beam of light passing through quartz, and probably due to the light scattering effect of extremely fine blue needles.

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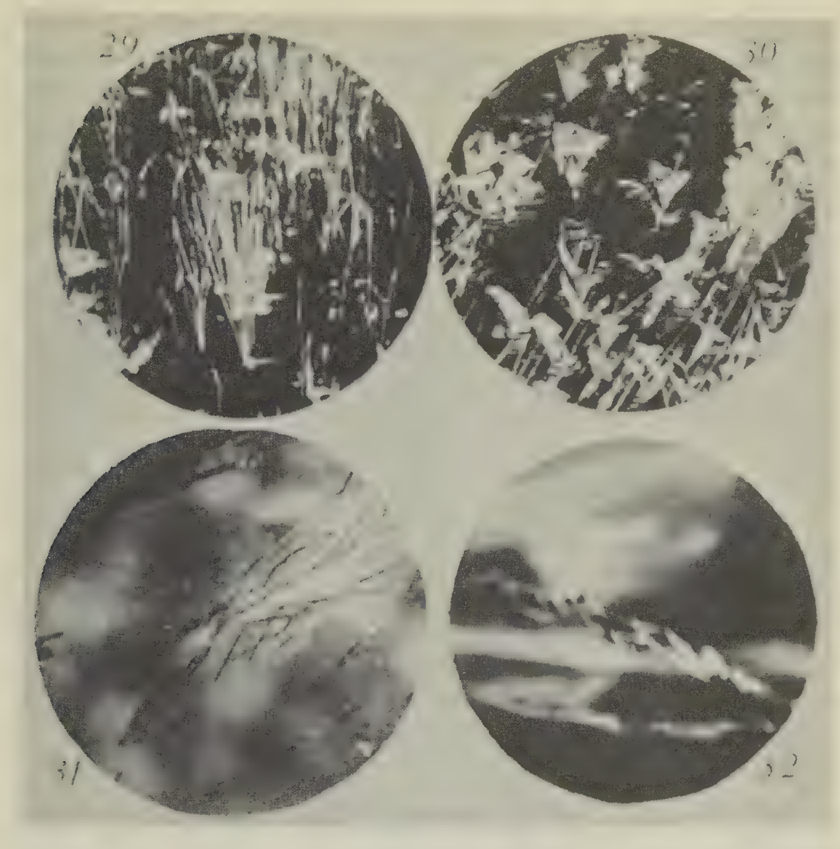
[12] This is obvious from the angle of 45° shown by the V's of fig. 27 as projected on the plane of the photograph.

[13] Willard, G. W., Raw quartz, its impurities and inspection: *Bell System Technical Jour.*, **22**, 338–361 (1943); *Mineral. Abstracts*, **9**, 83 (1944).



Figs. 29–32. Photomicrographs by Dr. Thomas S. Stewart (magnification 7×, 7×, 22×, and 70× respectively), of a wafer which had been etched. It is assumed that the etching fluid has proceeded along "blue needles" which occurred in the quartz, because of the V-shaped clusters shown (Fig. 29). Fig. 30 shows the limbs of the V proceeding from the edges of the triangular-pyramidal etch pits on the opposite side of the wafer. Fig. 31 emphasizes how the solutions followed parting planes in the crystal. Fig. 32 shows the ribbon-like, linear character of the etched planes; under 150× magnification one can see optical twin boundaries on these planes!

Grading Quartz

To justify cutting, a quartz crystal must have some of its space free of twinning and detrimental imperfections. Its value will depend on how much of it is truly flawless. Efforts have therefore been made to grade quartz on the basis of its *usability:* defined as the estimated partial volume that might yield oscillator-plates.

Grading has heretofore been limited to the results obtained in the oil bath: on the basis of the amount of optical twinning found, and of such imperfections as is revealed in the intense beam of a spotlight. No effort has been made to grade quartz on the basis of the amount of electrical twinning present, since this would require etching of the crystals—and dealers object to anything being done to their quartz—in fact, the oil bath has already been too revealing.

Weight classification: Quartz is sold by weight, and is sorted into weights of 100 to 200 grams, 200 to 300 grams, 300 to 500 grams, etc. In the Bureau of Standards code, the numbers 1, 2, 3, etc. are used to designate the lower limit in a box of quartz.

Faced quartz refers to material having at least one identifiable prism or rhombohedral plane measuring approximately $\frac{3}{4}$ inch square or more. Boulders and defaced quartz are called unfaced quartz.

Optical quartz includes flawless material, 500 grams or more in weight, free of strain, but not necessarily free of electrical twinning. There are two grades of usability, 45–60% and 60–100%.

Oscillator quartz is graded[14] according to the estimated percentage of usable volume, as well as for the presence or absence of structures or defects that might limit its use for some purposes. Grade 1 includes flawless material. Grade 2 contains as defects only blue needles, color or Tyndall effect. Grade 3 shows in the eye-clear portions some parting, bubbles, and inclusions.

Grades 1 and 2 are graded further according to percentage of usability, with limits of 30–45%, 45–60%, and 60–100% (or roughly into more than a quarter, a half, or three-quarters usable). Grade 3 was classified into two grades of "usability" of 0–45% and 45–100%.

Inspection During Processing

There are many steps in the reduction of a quartz crystal into the tiny oscillator blanks: the quartz must first be sliced into wafers, or if large, into sections, and then into bars suitable for wafering. More than 95% of the quartz is useless or wasted in the processing, and the final oscillators may represent less than 1% of the volume of the quartz that one started with. Grading of raw quartz is necessary to be sure that the quartz has flawless areas that justify its processing. When the quartz has been cut into sections, bars, and wafers it is the practice to etch them, and reject hopelessly twinned areas. The most important stages of inspection, however, are those of the blanks prior to lapping and final finishing. If cut from bars they should be etched and examined for twinning; it is assumed that only untwinned blanks were diced from etched wafers. Prior to lapping, the blanks must be subjected to rigid inspection, while immersed in oil, in an intense spot light, for bubbles, cracks, inclusions, and other defects (Figs. 33–35). These defects, as well as twin boundaries, are areas of weakness, and the blanks are liable to crack in

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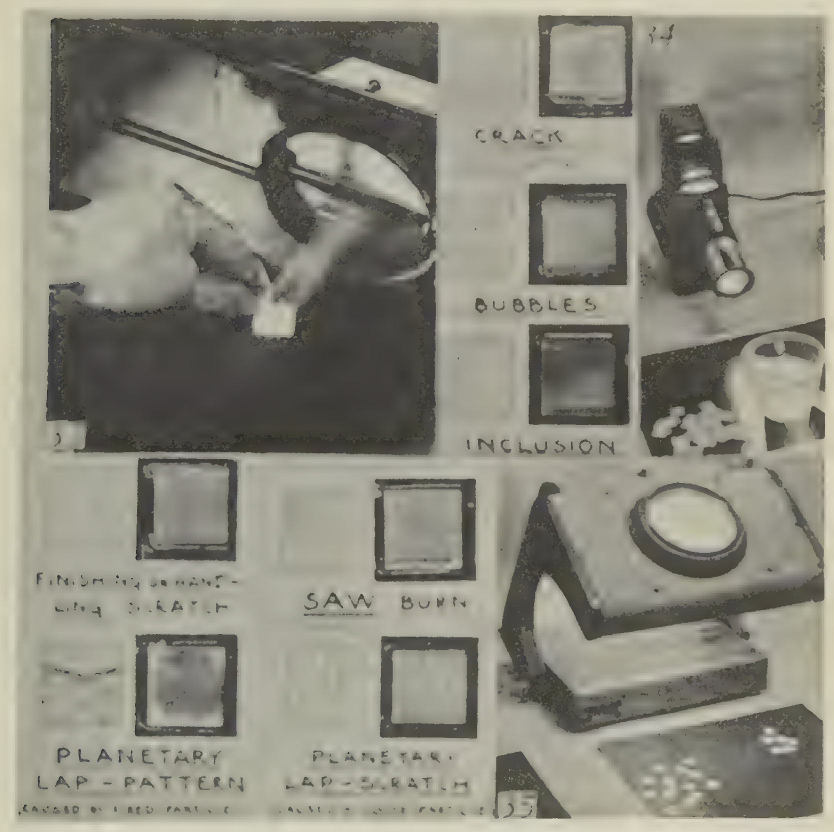
[14] National Bureau of Standards, Optics Division (FJB:MLH, IV-w. Ipp-941-c) "Specifications for testing and grading crystalline quartz in conformity with the conditions imposed by the procurement, purchase, distribution, and use of the material as found by the Board of Economic Warfare, Metals Reserve Company, War Production Board, and the U. S. Army Signal Corps." Washington, D. C. 1943.



FIGS. 33–35. Among the most important stages of inspection in processing are those of the blanks prior to lapping and prior to final finishing. Prior to lapping, blanks are inspected for internal imperfections (cracks, bubbles, inclusions) by immersing the blanks in oil and scanning them in an intense spot-light (Figs. 33–34). Prior to etching to frequency, lapped blanks are inspected in a powerful light for surface imperfections (scratches, chipped edges) (Fig. 35). (Blanks and drawings from a chart by Caroline Briggs and E. J. Korda (NAP).)

the laps—perhaps resulting in total loss of a lap load. Moreover, their rejection at this point eliminates lapping of useless material.

Lapping of blanks causes submicroscopic shattering of the surface to a depth of .25 to 0.5 microns. This shattered surface tends to disintegrate, and often, in the case of small, high frequency plates, results in total loss of activity. This deterioration, which has been dignified by the term "ageing," can be eliminated by dissolving the unstable, shattered surface. It is now, therefore, the practice to etch plates to their final frequency. Prior to such etching, the surface of the blanks must be examined in a strong spot-light to eliminate those that were scratched, since the etching solutions tend to attack those places, resulting in plates with lower activity.

THE EFFECT OF IMPERFECTIONS ON THE USABILITY OF QUARTZ FOR OSCILLATOR-PLATES

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ABSTRACT

Imperfect quartz used for the manufacture of communications crystals compared favorably with highest quality raw material in regard to yield of satisfactory oscillator-plates. Inclusions not visible except by their scattering effect on light do not prevent the use of quartz for crystals up to 8.5 megacycles. Inclusions visible to the unaided eye reduce ability of the quartz to oscillate. Some twinning may be tolerated in specialized locations.

INTRODUCTION

During the winter of 1942–1943, the United States was threatened with a serious shortage of imported quartz. Since domestic production was negligible, it was necessary for the Signal Corps to consider methods of expanding the usefulness of the existing stock-pile. One possibility lay in the use of raw material which had been classified as below grade and set aside. Some 500,000 pounds had been so classified, principally because of the presence of blue needles. Since no real evidence existed as to whether the presence of blue needles—or, indeed, any other of the recognized flaws—rendered the quartz unusable for oscillator-plates, an investigation was initiated to determine the usability of imperfect quartz for AT- and BT-cut plates of frequencies up to 8500 kc/sec. The study reported herein was the confirming evidence which brought about the revision of specifications and the release of the greater part of the so-called "below grade" quartz for use in the manufacture of quartz plates.

THE NATURE OF IMPERFECTIONS

In an accompanying paper, Gordon[1] has described the nature of the various imperfections found in quartz. They may be divided into two groups: inclusions and structural defects. Inclusions range in size from atomic impurities through needles visible under ordinary lighting. This category also includes bubbles, which may be considered to be negative inclusions. Very small inclusions are visible only through their scattering effect on an intense beam of light. The principal structural defect is twinning, although lineage structure is present.

The experimental work described in this report was concerned primarily with the inclusion imperfections. They were classified, not on the basis of their mineralogical character, but, rather, in the categories recognized by raw quartz inspectors. This resulted in duplication as, for instance, veils are merely microscopic bubbles localized in curved sheets. Phantoms are either color or bubbles, or combinations of both, parallel to natural crystal surfaces.

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[1] Gordon, Samuel G., The inspection and grading of quartz. *Am. Mineral.*, this issue.

INCLUSIONS

Selection of Specimens. The majority of the inclusions are difficult to locate in a finished oscillator-plate and, in order to be sure that the plates were representative, it was necessary to choose raw material with high concentrations of each imperfection. Since, as a rule, the imperfections are not uniformly distributed throughout a given piece of quartz, selected specimens were trimmed of all material not containing the imperfection being studied. Because of this no data can be given on yield per pound of raw material. Samples were selected which contained as high a concentration as could be found of the following imperfections: smokiness, blue needles, chuva, rutile needles, microscopic bubbles, veils and phantoms. Every effort was made to obtain material containing only the particular flaw being investigated.

Preparation of Oscillator-Plates. The specimens containing each imperfection were processed into oscillator-plates following the usual manufacturing techniques. That is, no attempt was made to make the plates to any predetermined dimensions. Each plate was hand lapped to final frequency and edge-ground to adequate activity. Frequencies ranged from 3500 kc/sec to 8500 kc/sec. The finished plates were then mounted in holders and tested for performance over a temperature range of −35°C to +55°C. Control crystals were also prepared from clear quartz of Grade A quality.

Performance tests consisted of measuring frequency and activity over the prescribed temperature range, using the standard tolerances applicable in certain Signal Corps radio sets. The allowable frequency drift was plus or minus two hundredths of a per cent. There were no failures because of excessive frequency drift, nor was there any evidence of influence of imperfections on the frequency characteristics, within limits of measurement.

In all cases, failure of a plate to meet specifications was due to low activity at some point in the temperature range. Such "activity dips" are common-place in plates made without regard to pre-determined lateral dimensions. The usual procedure is to disassemble the crystal unit and adjust the lateral dimensions by edge grinding until the "dip" has moved out of the specified temperature range. In this investigation such re-working of the plates was not carried out. Hence, failure of an oscillator-plate to meet activity requirements does not mean, necessarily, that it could not be made acceptable. The purpose of this study was to determine the comparative yields obtained from clear, Grade A quartz and from imperfect material. It was sufficient to compare only the initial yields.

Experimental Results. In Table 1 are listed the experimental results for oscillator-plates made from quartz containing the various inclusions. In addition to those listed in the table, a number of plates were made from smoky quartz of varying intensity of color. It was found to be no more difficult to process colored quartz than clear quartz except for optical orientation of very dark material. Finished plates exhibited the same physical properties—frequency-thickness constant (within limits of measurement), characteristic shape of frequency vs. temperature curve, activity dips, etc.—as clear quartz.

TABLE 1

Inclusion	Number of Plates Processed	Per Cent Meeting Specifications
Blue needles	149	72.5
Chuva	83	73.5
Visible rutile needles	52	0.0
Microscopic bubbles	143	66.5
Veils	104	64.5
Phantoms (color and bubbles)	83	83.0
Total (less visible rutile needles)	562	71.2
Control (Grade A quartz)	133	70.6

Discussion. The data listed in Table 1 indicate that those inclusions which are visible only by their scattering effect on a strong beam of light do not seriously affect the use of quartz for communications crystals. The same is true of color, which may be considered as being due to inclusions of atomic size. Rutile needles which are visible under ordinary lighting conditions prevent or decrease oscillation. It would appear from these results that size is the determining factor, rather than the nature of the inclusion. Blue needles, for instance, are generally supposed to consist of rutile needles of the proper size to scatter light with a Tyndall effect. Chuva is believed to be, in part, somewhat larger rutile needles. A thorough investigation, which was beyond the scope of this work, would probably reveal a relation between the fundamental frequency of the oscillator-plate and the permissible size of inclusions. Thus, in very high frequency plates, up to 25 megacycles per second, small inclusions seem to have a definite deleterious effect.[2] Similarly, low frequency crystals, particularly filter crystals, can tolerate substantial amounts of relatively large inclusions.

STRUCTURAL DEFECTS

Twinning. The commonest imperfection in quartz is twinning. Two twin laws predominate and these occur to such an extent that it is an extremely rare crystal that does not exhibit both. One of the types, electrical, has the effect of reversing the polarity of the two-fold axis, the handedness of the individuals remaining the same. The other, optical, is characterized by right- and left-handed individuals, as well as reversed polarity of the axis. Obviously, a satisfactory oscillator-plate could not be made if there were any great percentage of the quartz twinned as the individuals would have opposite electrical charges. Optically twinned quartz would be more unsatisfactory since the individuals of the twin would have different frequencies. However, it is not uncommon to find small areas of either electrical or optical twinning in oscillator-plates which have passed all performance tests. The areas must be small and should be confined to the corners and possibly the edges. In Fig. 1 are sketched three oscillator-plates which have adequate activity even though they contain twinning. Both optical (O) and electrical (E) twinning are present in (a) with only optical in (b) and (c). It is to be noted that the twinned areas can be quite large when they are located at the corners. However, that in the center (c), is only a small fraction of the area. Doubtless it would be possible to determine the amount and areal distribution permissible for each type of twinning. However, it would be impractical from a production point of view to produce plates in this

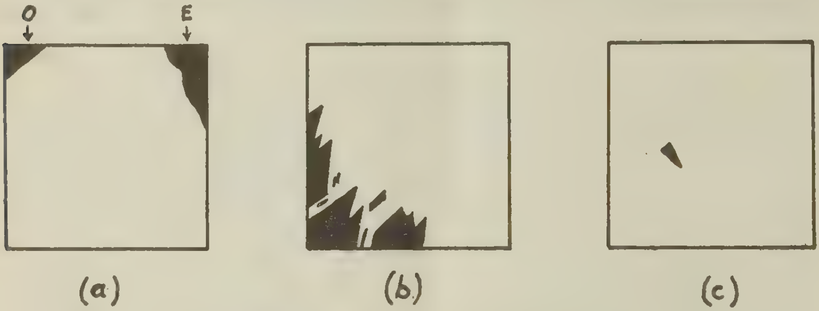

FIG. 1. Oscillator-plates with areas optically and electrically twinned.
1(a) Optical (O) and electrical (E) twinning.
1(b) and (c) Optical twinning.

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[2] Frondel, C., personal communication.

manner. The same should be true of rutile needles and large bubbles. These latter two are probably permissible in specialized locations.

Lineage Structure. The manufacture of oscillator-plates for communications purposes does not require extreme delicacy in physical measurements. For this reason, lineage structure has presented no problem. The effect of lineages—or discrete blocks, if such are present—would be to lower the Q of the plate. That is, there would be a damping effect due to the structural imperfection. The amount of damping could probably be measured under laboratory conditions, but its effect on the so-called "activity" of the oscillator-plate would not be noticeable in communications equipment. Reported occurrences of inactive or weakly active quartz may be the result of extreme lineage structure or of microscopic twinning.

PRECISION CRYSTALS

The experimental results discussed above can be considered applicable only to plates produced by the usual technique whereby adequate activity over the temperature range is achieved by edge-grinding each oscillator-plate individually, so that no interfering modes of oscillation are coupled to the main mode throughout the applicable temperature range. Ideally, the proper dimensions should be determined for each frequency in advance and all oscillator-plates made to those dimensions. Although no thorough investigation was made of the possible effect of the various imperfections on the validity of pre-determined dimensions, encouraging indications that the dimensions will still be valid were obtained from studies of a precision crystal. Several oscillator-plates to be used in a 1000 kc/sec calibrating crystal unit having very close frequency drift tolerances were made from quartz containing blue needles. Dimensions which had been derived for clear, Grade A, quartz were used. All of the oscillator-plates met the specifications. Another indication that imperfections do not seriously affect the validity of pre-determined dimensions can be gathered from the fact that most dimensions being used at the present time are determined without regard to imperfection content. The usual yield of good plates is in the neighborhood of ninety-five or more per cent. Some of the failures may be attributable to imperfections, but the percentage is extremely small.

It is probable that any twinned areas in an oscillator-plate would have a noticeable effect on the validity of pre-determined dimensions. Such areas would effectively change the dimensions, so that the operating dimensions would not be the actual physical ones of the plate. In this connection, twinning on a microscopic or smaller scale would have the same effect and might account for some failures of otherwise correctly dimensioned plates.

ORIENTATION TECHNIQUES FOR THE MANUFACTURE OF QUARTZ OSCILLATOR-PLATES

William Parrish* and Samuel G. Gordon†

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Abstract

This paper describes the theory, methods and equipment used in orienting raw quartz, and the reorientation of sections, bars, wafers and blanks in various stages of processing. The orientation of quartz by optical determination of its "hand" and electrical measurement of polarity of the electric axis is discouraged because the latter test may be completely misleading due to electrical twinning. The characteristic light figures, obtained on etched sections and bars when placed over an illuminated pin-hole, are the sole infallible and unambiguous criteria for the orientation of quartz. The light figures on etched Z- and X-sections and the determination of usable portions of electrical twins in etched wafers by characteristic light reflection or transmission are described. The optical methods include the use of two simple types of stauroscopes: one for orienting Z parallel to the reference edge of a glass mounting plate, and the other for determining X and Z directions in wafers. The conoscope is valuable for locating Z in quartz whether faced or defaced. The notation

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of axes, faces and cuts is given; all ambiguity is removed by referring the cuts to the crystal faces. Various minor methods such as the development of parting parallel to the X-axes by slowly immersing a heated thin basal section in water are reviewed.

Introduction

The quartz oscillator, as used for frequency control in radio and telephone communication systems, is one of the most precise devices developed by modern science.[1] Resembling small square microscope cover glasses, plates of one frequency may differ in thickness from plates of adjoining channels by as little as one ten-millionth of an inch. Theoretically, a difference of ten cycles, which is easily measurable, means a reduction in thickness of but one layer approximating a unit cell in thickness, in the case of an 8 Mc/sec BT crystal.

The quartz plates used by MM. Jacques and Pierre Curie in their discovery of the piezoelectric effect were "X-cuts" with their largest surface perpendicular to the electric axis, X (the crystallographic axis *a*), length parallel to the mechanical axis, Y, and width parallel to the optic axis, Z (crystallographic axis *c*).[2] Such a plate, parallel to $(11\bar{2}0)$, could be cut by simply laying a crystal on its prism face (m $10\bar{1}0$), and sawing parallel to the vertical optic axis (Z, *c*); it was not necessary to determine the hand of the crystal or polarity of the X-axis.

Although X-cuts are still used in ultrasonic devices and other special applications where frequency change with temperature is of no importance, or may be controlled by the use of thermostatically controlled ovens, the vast majority of the crystals manufactured today are of the low temperature coefficient type.[3] These crystals are cut at selected pre-

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[1] In a paper by C. F. Booth, The application and use of quartz crystals in telecommunications: *Jour. Inst. Elect. Eng.*, **88**, 97–144 (1941), Mr. A. J. Gill states (p. 129) "the author mentions that the error of a certain crystal standard is ±0.00086 sec. in 24 hours; this means that a clock driven from this frequency standard would gain or lose less than 1 sec. in 3 years, and in this period the crystal will have made 10^{14} vibrations."

[2] Curie, Jacques et Pierre, Developpement, par pression, de l'electricité polaire dans les cristaux hemiedres à faces inclinées: *Compt. Rend.* **91**, 294–295 (1880); Sur l'electricité polaire dans le cristaux hemiedres à faces inclinées: *ibid.*, **91**, 383–386 (1880); Contractions et dilatations produites par les tensions electriques dans les cristaux hemiedres à faces inclinées: *ibid.*, **93**, 1137–1140 (1881). Friedel, C., and Curie, J., Sur la pyroélectricité du quartz: *Bull. Soc. franc. Min.*, **5**, 282–296 (1882).

[3] The Coast Guard desiring to use quartz crystals to control radio communications from the cold of Alaska to the warmth of Florida requested more stable plates from the Western Electric Co. a little more than a decade ago.

More than 95% of the crystals manufactured today are AT-(1 to 5.5 MC), BT-(5.5 to 9 MC) and CT-(200 to 500 KC) cuts. For a description of the properties of these and other types of crystals see: Mason, W. P., Low temperature coefficient crystals: *Bell System Tech. Jour.*, **19**, 74–93 (1940); Lack, F. R., Willard, G. I. and Fair, I. E., Some improvecise angles to the optic axis but with one edge parallel to the electric axis (except the GT-cut) and do not require temperature control to keep military communications within their specified channels. The most commonly used cuts and their orientations are shown in Fig. 1. Temperature control is required only where accuracy of the order of a few parts per million must be maintained as in commercial broadcast stations, frequency standards, etc.

Radio engineers defined the cuts with respect to the electrical axis (X) and the optic axis (Z). A quartz crystal was squeezed between two opposite prism edges (from which the X-axis emerges) and the reaction noted on an electrometer or similar device. If the polarity of the edge under test was positive on compression, the AT-cut was made at an angle of +35°15′ (clockwise) from the optic axis in a right-hand crystal, and the BT-cut at an angle of −49° (counterclockwise) from the optic axis. The directions were reversed for a left-hand crystal. A polariscope was used to determine the hand of the crystal.

The usage of the terms right- and left-hand by radio engineers has been generally the *reverse* of that of crystallographers and mineralogists. In using the Herschel convention the radio terminology has followed that employed by chemists in polarimetry and saccharimetry in which a quartz crystal is called right-handed if it rotates the plane of polarization to the right (clockwise) when the eye is following the light rays. The confusion has arisen from the apparent reversal of the direction when observed with the eye adjacent to the crystal looking toward the oncoming rays (Biot convention). A discussion of the confusion in the nomenclature of rotatory power is given by Sosman.[4] All ambiguity is removed if the cuts are related to the crystal faces.[5]

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ments in quartz crystal circuit elements: *ibid.*, **13**, 453–463 (1934). Low temperature coefficient crystals were also developed independently at about the same time in Germany and Japan: Bechmann, R., Temperature coefficients of vibrations of quartz plates and bars: *Hochfreq. Tech. u. El. Ak.*, **44**, 145–160 (1934); Straubel, J., *Zeit. tech. Physik.*, **35**, 179 (1934); Koga, I., Thermal characteristics of piezoelectric oscillating quartz plates: *Report of Radio Researches and Works in Japan*, **4**, 61 (1934). See also series of papers in *Bell Syst. Tech. Jour.*, **22** (1943), **23** (1944) and *Bell Lab. Record*, **22** (1943–44).

The GT-cut crystal, ZZ′ = +51°30′ with its edge rotated 45° from the X-axis, has an extraordinarily low temperature coefficient. Used for low frequencies (100 KC), it is constant to one part in a million in the temperature range 0° to 100°C. and over a 30°C. region the frequency change is one part in ten million with no temperature control. Mason, W. P., A new quartz crystal plate, designated the GT, which produces a very constant frequency over a wide temperature range: *Proc. Inst. Radio Eng.*, **28**, 220–223 (1940).

[4] Sosman, Robert B., *The Properties of Silica*, The Chemical Catalog Co., Inc., New York (**1927**), pp. 648–649. *Dana's Textbook* and *Manual* use the Herschel convention in the optical parts of the books but the Biot convention is followed in the crystallographic

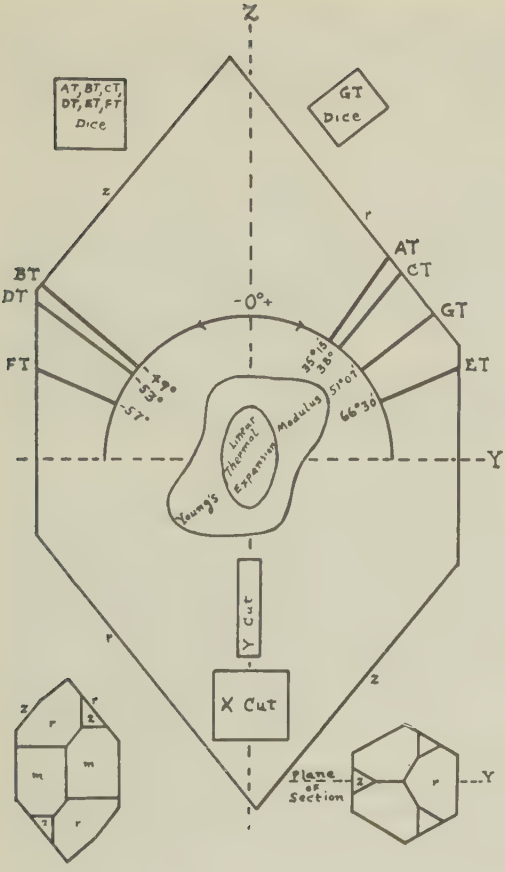

FIG. 1

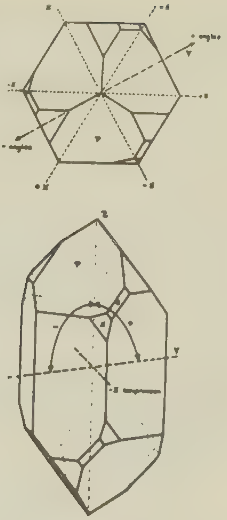

FIG. 2

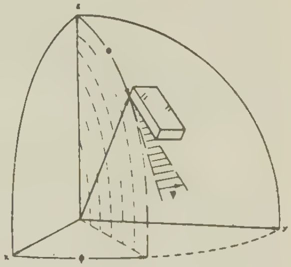

FIG. 3

FIG. 1. Orientation of principal types of oscillator-crystals and their cutting angles. AT, CT, GT and ET approximately parallel to minor rhomb *z*; BT, DT and FT approximately parallel to major rhomb *r*. X parallel to edge of plates except GT which is rotated 45°. X- and Y-cuts are perpendicular to X- and Y-axes, respectively.

FIG. 2. The principal axes of quartz. $Z=c=$optic axis; $X=a=$electric axes; $Y=$mechanical axes. (+) direction from Z is rotation towards parallelism with *z* and (−) direction towards parallelism with *r* regardless of hand of crystal. (+) is clockwise in right-hand crystal and (−) counterclockwise but are reversed in left-hand crystal. In an untwinned crystal the ends of the X-axis on a prism edge containing the *s* and *x* faces become negatively charged on compression and positively charged on release of compression (tension or stretching) by application of a stress along X.

FIG. 3. I.R.E. Orientation angles. ϕ measured from X $(a)=$complement of crystallographer's ϕ; θ measured from Z $(c)=\rho$ of crystallographers; $\psi=$dicing angle. Drawing from W. L. Bond, *Bell Syst. Tech. Jour.*, **22**, 224 (1943).

The manufacture of quartz oscillator-plates involves not only the orientation of the "raw" quartz, usually known as "mothers" in the industry—whether faced or completely defaced, but the reorientation of sections, bars, wafers, or blanks in various stages of processing, and the recognition of electrical and optical twinning in this material—and particularly the orientation of the latter in the wafers under examination. Moreover, the angular deviations from specified tolerances must be accurately determined and controlled. While this seems a formidable task, it may be readily accomplished from the morphology of the quartz by the unique properties along the optic axis, the etch patterns shown in various sections, or by measurement of angles from reference atomic planes within the crystal by means of x-rays. The use of the x-ray method requires prior orientation by some of the methods described here, before it can be successfully applied, and is described in the following paper.[6]

This paper deals only with certain technological aspects of the practical problems closely related to the manufacture of quartz oscillator-plates. A portion of this subject matter especially prepared for crystal manufacturers has been published by the Signal Corps.[7] Time and space requirements do not permit the presentation of crystallographic data collected on etch figures, x-ray reflections, inclusions and a host of other interesting phenomena. For the same reasons, the literature cited is not a complete bibliography. It is our desire to present these supplementary data at the earliest possible date.

Acknowledgments. The writers are indebted to Mr. John Derbyshire, N.A.P., for taking many of the photographs and Mr. Charles E. Goldmann, N.A.P., for converting the stauroscopes illustrated, from commercial instruments. The conoscope was designed by Mr. W. L. Bond of Bell Telephone Laboratories. Figures 3 and 12 are from Bond, *Bell*

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description of quartz. Other inconsistencies have been pointed out by K. S. Van Dyke, On the right- and left-handedness of quartz and its relation to elastic and other properties: *Proc. Inst. Radio Eng.*, **28,** 399–406 (1940). The adaption of the Biot convention which makes a given crystal either right- or left-handed in both the commonly used crystallographic and optical descriptions is recommended also to radio engineers by W. G. Cady and K. S. Van Dyke, Proposed standard conventions for expressing the elastic and piezoelectric properties of right and left quartz: *ibid.*, **30,** 495–499 (1942).

[5] The Japanese have defined cuts by their angles from the major and minor rhombohedral faces.

[6] Parrish, William and Gordon, Samuel G., Precise angular control of quartz cutting with x-rays: *Am. Mineral.*, this issue.

[7] Parrish, William and Gordon, Samuel G., *Manual for the Manufacture of Quartz Oscillator-Blanks*, Office of the Chief Signal Officer, War Department, Washington, D.C., 1943. Note: this publication is no longer classified as *restricted*.

Syst. Tech. Jour., **22**, 224 (1943). Figure 4 showing the Airy's spirals is from C. F. Booth, *Jour. Inst. Elect. Eng.*, **88**, 97 (1941). Mr. John D. Davies, N.A.P., aided in preparation of the section on electrical polarity determination.

Notation of Axes, Faces, and Cuts

The optic axis (crystallographic axis c) is conventionally designated Z. The horizontal crystallographic axes a_1, a_2, and a_3, are called the electric axes, each designated by the letter X. The three intermediary, horizontal axes, each at right angles to an X axis, are called the Y axes, or mechanical axes (Fig. 2).

The striations on the prism faces m $(10\bar{1}0)$ are parallel to the X axes, which emerge at the prism edges (normals to $11\bar{2}0$). The normals to the prism faces m $(10\bar{1}0)$ are the Y axes. Due to the oscillatory development of the prism faces (between the prism and terminal rhombohedrons), the prism edges are but rarely parallel to the optic axis.

The terminal rhombohedrons, r $(10\bar{1}1)$ and z $(01\bar{1}1)$ are distinguished as the *major* and *minor* rhomb faces, respectively.

The modifying planes s $(11\bar{2}1)$ the right trigonal pyramid, and x $(51\bar{6}1)$ the right positive trapezohedron, form a zone with m $(10\bar{1}0)$ and z $(01\bar{1}1)$, to the right of m $(10\bar{1}0)$ and serve to identify such a crystal as right-handed (Fig. 5). The left trigonal prism $'s$ $(2\bar{1}\bar{1}1)$ and the left positive trapezohedron $'x$ $(6\bar{1}\bar{5}1)$ form a zone with m and z to the left in left-hand crystals (Fig. 4).

A right-hand crystal appears to rotate the plane of polarization to the right (clockwise) on looking towards the light source (Biot convention).

It will be noted that there are six major rhombohedral faces (r), three at each end of the crystal; and likewise six minor rhombohedral faces (z) at each end of the crystal. For each r face, there is another r face—opposite and parallel to it on the other end of the crystal. Likewise, each z face is opposite and parallel to another z face. Thus there are three pairs of parallel major (r) rhomb faces, and between them three pairs of minor (z) rhomb faces.

The AT-, CT-, ET-, and GT-cuts may be thought of as made between a pair of minor (z) rhomb faces, and not far in angle from parallelism with face z.

The BT-, DT-, and FT-cuts may be considered as made between a pair of major (r) rhomb faces, and not far in angle from parallelism with face r.

The "I.R.E. Orientation Angles." A committee of the Institute of Radio Engineers has recommended the adoption of the following method of specifying the orientation of a quartz plate. In this new system, a cut is defined with respect to three axes at right angles to each other: Z (the *c* or optic axis), X, the electric axis (the *a* axis normal to $11\bar{2}0$), and Y, the mechanical axis (normal to *m* $10\bar{1}0$) as shown in Fig. 3. It will seem strange to crystallographers that three angles are necessary to define a

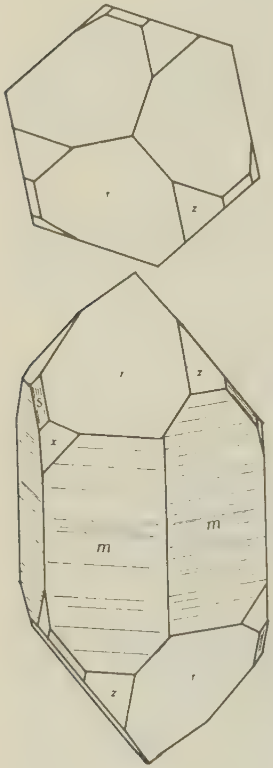

Fig. 4

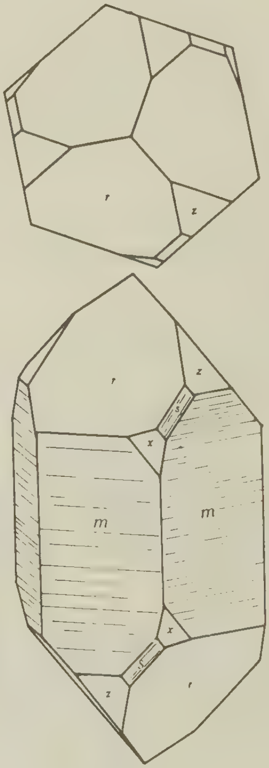

Fig. 5

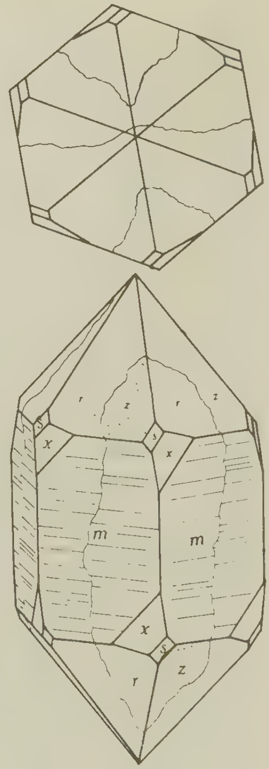

Fig. 6

Fig. 4. Left-hand quartz. With Z (*c*) vertical, '*s* ($2\bar{1}\bar{1}1$) and '*x* ($6\bar{1}\bar{5}1$) form a zone with *m* ($10\bar{1}0$) and *z* ($01\bar{1}1$) to the *left* of *m*. Rotation of plane of polarization is to the *left, counterclockwise*, on looking *toward* source of light (Biot convention).

Fig. 5. Right-hand quartz. With Z (*c*) vertical, *s* ($11\bar{2}1$) and *x* ($51\bar{6}1$) form a zone with *m* and *z* to the right of *m*. Rotation of plane of polarization to the *right, clockwise* (Biot convention).

Fig. 6. Electrical twinning (Dauphiné law) of left-hand individuals. The individuals are of the same hand and related by a 180° rotation about Z (*c*) so that the crystallographic axes are parallel but the polarities of the electric axes X (*a*) are reversed and *r* and *z* become coplanar. Electric polarity determination of front edge of crystal would be misleading for orientation purposes due to electrical twinning.

plane but this is true of only two of the angles ϕ and θ. ϕ is measured from the electric axis (crystal axis a) and is therefore the complement of ϕ of crystallographers. The angle θ corresponds to the ρ angle of crystallography. The third angle ψ, is merely the dicing angle, the angle which the long edge of the blank makes with the trace of the meridian ϕ.

Cut[8]	ϕ	θ	ψ
z	0°	0°	0°
x	0	90	90
y	90	90	90
AT	−90	$54\frac{3}{4}$	90
BT	−90	−41	90
CT	−90	52	90
GT	−90	38°52′	±45
NT	6°40′	50°28′	79°36′

Radio engineers often refer to the ZZ′ and XX′ angles. ZZ′ is the angle between the optic axis, Z, and the plane of the blank, Z′. XX′ is the angle between the electric axis, X, and the edge of the blank, X′.

Electrical Determination of Polarity of X-axis

The polarity of the electric axis X may be determined by applying pressure across opposite prism edges and noting the polarity of the voltage produced with a suitable electronic circuit. In quartz it is a rapid *change* in pressure which produces the voltage. The polarity on tension (release of pressure) is the reverse of that obtained on compression. *In an untwinned left- or right-hand crystal, that end of the X-axis which emerges at a prism edge below the modifying s and x faces becomes negatively charged on compression.* This is consistent with the kind of charges developed on this edge on *cooling* a quartz crystal.[9] Here the temperature *gradient* is due to the inside of the crystal being at a higher temperature than the surface. The temperature gradient is reversed on heating the crystal and the charges developed have the opposite sign.[10] The pyroelectric test may be tried by Kundt's method using charged red lead and sulfur powders[11] or the magnesium smoke[12] or liquid air methods[13] but has not been employed as a production technique in this country.

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[8] Bond, W. L., Method for specifying quartz crystal orientation and their determination by optical means: *Bell Syst. Tech. Jour.*, **22,** 224–262 (1943).

[9] Von Kolenko, B., Die Pyro-Elektricitat des Quarzes in Besuch aus sein kristallographisches System: *Zeit. Kryst.*, **9,** 1–28 (1884).

[10] Wooster, W. A., *A Text Book onCrystal Physics*, Cambridge University Press, Macmillan, New York (**1938**), Chap. VII, Pyro-Electricity.

Several types of devices have been developed to determine the piezoelectric effect. Where quantitative measurements are not required the polarity indicator shown in Fig. 7 may be employed. It gives the polarity of the X-axis which emerges at the edge which is *down* on the lower platform. A rapid *change* in pressure produces a voltage which is impressed on the grid of a high gain amplifier such as the 6J7. A negative charge impressed on the grid will decrease the plate current and a positive charge will cause an increase in plate current. A milliammeter in the plate circuit of the 6J7 permits visual indication of the polarity. The plate current can be adjusted to one-half full scale value on the meter thus allowing + or − readings on the same meter. The meter deflection is observed immediately on applying pressure by means of the arm; it is reversed on releasing pressure.[14]

ORIENTATION BY MORPHOLOGY

In a well developed crystal it is easy to recognize the optic axis, (*c*, Z) which coincides with the principal crystallographic axis; the electric axes (X) $[11\bar{2}0]$ which are also the striations on *m* $(10\bar{1}0)$ and the mechanical axes (Y) which are the normals to the prism faces (*m* $10\bar{1}0$).

More difficult, however, is the distinction between the rhombohedral planes *r* $(10\bar{1}1)$, the major rhomb; and *z* $(01\bar{1}1)$, the minor rhomb; and since Dauphiné (electrical) twinning, in which *r* and *z* are coplanar is so common, the effort is futile. Much faith has been placed on determinations based upon the relative size of the *r* and *z* faces, and in the general rule that in "candles" the prism faces become narrower below *r* faces. Where triangular etch pits occur on the prism faces, the apices of the triangles always point to the major (*r* $10\bar{1}1$) faces (Fig. 8). Striations when discernible on *s* $(11\bar{2}1)$ are parallel to the major rhomb *r* $(10\bar{1}1)$ (Fig. 8). Crystals which have adjacent terminal faces of the same size and those in which the edges of adjacent terminal faces are parallel are almost certainly twin crystals.

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[11] Kundt, A., Ueber eine einfach Methode zur Untersuchung der Thermo-, Actino- und Piezoelectricitat der Krystalle: *Ann. Phys. u. Chem.*, **20**, 592–601 (1883).

[12] Maurice, M. E. On the demonstration of electric lines of force, and a new method of measuring the electric moment of tourmaline: *Proc. Camb. Phil. Soc.*, **26**, 491–495 (1930).

[13] Bleekrode, L., Über einige Versuche mit flüssiger Luft: *Ann. Phys.*, **12**, 218–223 (1903). Martin, A. J. P., On a new method of detecting pyro-electricity: *Mineral. Mag.*, **22**, 519–523 (1931).

[14] Electrical twinning in unetched quartz sections was once determined with an apparatus for detecting polarity with a probing electrode. A grid was ruled on the section, or illuminated from below, the surface was probed and the resulting polarities contoured on the grid!

The zone of minor faces x $(51\bar{6}1)$, s $(11\bar{2}1)$ and z $(01\bar{1}1)$ are to the right of r $(10\bar{1}1)$ in a right-hand crystal, and to the left in a left-hand crystal.

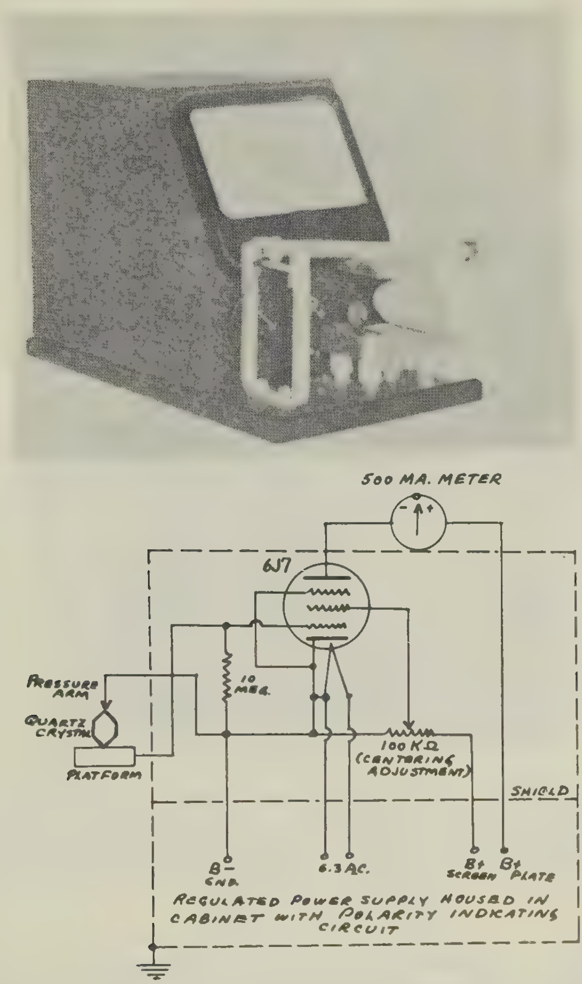
500 MA. METER
6J7
PRESSURE ARM
QUARTZ CRYSTAL
PLATFORM
10 MEG.
100 KΩ (CENTERING ADJUSTMENT)
SHIELD
B− GND.
6.3 AC.
B+ SCREEN
B+ PLATE
REGULATED POWER SUPPLY HOUSED IN CABINET WITH POLARITY INDICATING CIRCUIT

FIG. 7a. Piezometer for determining polarity of electric axes by squeezing crystal along X and noting meter deflection. Reaction must be noted immediately on squeezing and reverses on release of pressure. Fig. 7b. Circuit prepared by John D. Davies, NAP.

It is therefore necessary to supplement this observation with an optical test for chirality. Even if these faces are absent, the prism edge which would have been contiguous to them gives a negative reaction on compression on the piezometer, and thus serves as an aid in orientation. It must be emphasized that such a test is diagnostic only for the point squeezed, and if the quartz is electrically twinned, is ambiguous. If an electrically twinned crystal such as the one shown in Fig. 6 was oriented by testing the front side, the resulting wafers would be useless.

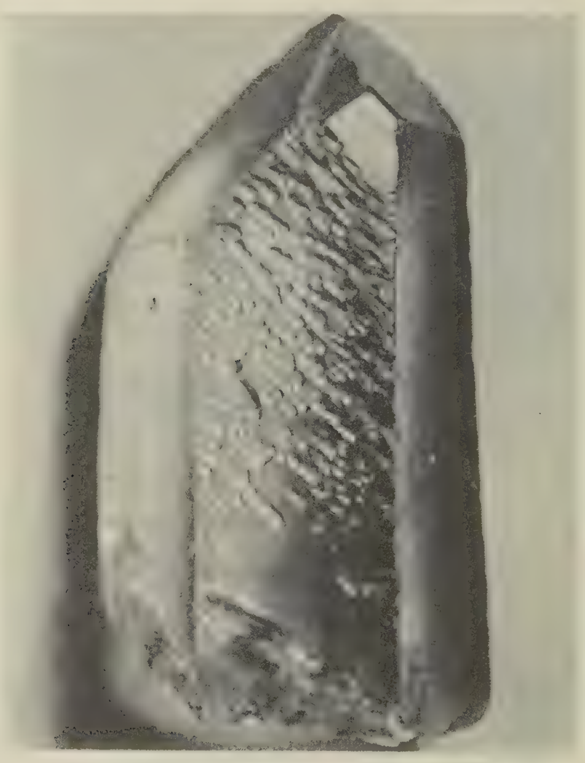

FIG. 8. Natural etch figures on Brazilian quartz. Apices of triangular pits on *m* $(10\bar{1}0)$ point to the major rhomb *r* $(10\bar{1}1)$, the largest apex face in the picture. The deep etch pits on this crystal occur on alternate prism faces, none occurring on the intermediate prism faces. The striations on *s* $(11\bar{2}1)$ are parallel to another *r* face in the upper right hand corner

ORIENTATION BY PARTING

Dana[15] lists as "cleavage: *r*, *z*, difficult and not often observed, also *m*, and sometimes *c*, more difficult; sometimes developed by sudden cooling after being heated; also by the pressure of a sharp point on thin sections, *e.g.*, cut $\parallel c$ and $\perp m$." This parting of quartz on *r* $(10\bar{1}1)$ is skillfully utilized in cobbing quartz in Brazil, and a very practiced eye can set up quartz by this means.

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[15] Dana, VI System, p. 186 (**1892**).

When a thin Z-section, 0.050″ to 0.100″ thick, is heated a few minutes on a hot plate and slowly immersed in a beaker of water at room temperature, prismatic cracks develop parallel to the X-axes (Fig. 9). The set of parallel, almost equally spaced cracks which first form are those more nearly perpendicular to the water surface. By slowly rotating the section while immersing, sets of cracks parallel to the other X-axes are developed. The cracks grow slowly at a rate dependent upon the rate of immersion, accompanied by a clearly audible cracking sound and their growth may be readily observed. The surfaces are remarkably straight and parallel to the X-axes and often follow a zig-zag path. The section is easily broken along the cracks and the surfaces have a brilliant luster. Rapid immersion and the use of thick sections generally causes a shattering and a multitude of random cracks. The method has been used abroad to determine X in defaced quartz by refitting the cracked section to the crystal.[16]

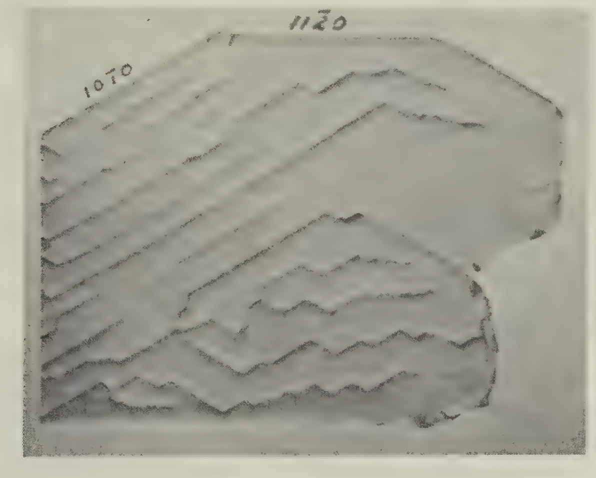

Fig. 9. Prismatic parting parallel to X-axes developed on 0.090″ thick heated Z-section slowly immersed in beaker of cold water.

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[16] Booth, C. F., *op. cit.* This type of thermal parting seems to have escaped the attention of mineralogists. See also Judd, J. W., On the development of a lamellar structure in quartz by mechanical means: *Mineral. Mag.*, **8**, 1–8 (1888); Mallard, E., Sur les clivages du quartz: *Bull. Soc. franc. Min.*, **13**, 61–62 (1890); Schubnikow, A. and Zinserling, K., Über die Schlag- und Druckfiguren und über die mechanischen Quarzzwillinge: *Zeit.Krist.* **8**, 243–264 (1932); Drugman, Julien, Prismatic cleavage and steep rhombohedral form in α-quartz: *Mineral. Mag.*, **25**, 259–263 (1939); Fairbairn, H. W., Correlation of quartz deformation with its crystal structure: *Am. Mineral.*, **24**, 351–368 (1939); Frondel, Clifford, Secondary Dauphiné twinning in quartz: *Am. Mineral.*, this issue.

Other methods of less value are no longer used.[17] In one, a small steel ball was dropped upon a basal section from a height of about half a meter. The section was polished and the sides of the dent were approximately parallel to the X-axes. The method has many obvious disadvantages as well as being inaccurate. Rivlin's method[18] consisted of grinding one side of a Z-section with coarse silicon carbide using a rocking and crunching motion. A diffuse hexagonal light figure with sides approximately parallel to the natural prism faces appears when viewed over a pin-hole source of light.

Optical Methods for Orientation of Quartz

The optical methods for quartz crystal orientation comprise the stauroscopic or conoscopic location of the optic axis in faced or defaced quartz, or in sawed material in the process of manufacture.

Mounting Stauroscope.[19] This instrument consists simply of a pair of crossed Polaroids, a source of illumination, and a stage with a reference edge, upon which a quartz crystal, section or bar can be oriented for mounting on a glass plate for sawing (Fig. 10). The approximate direction of the optic axis must be known from morphology of the crystal or by examination in the inspectoscope or conoscope. It is used only in lining up the optic axis of the quartz to about a degree of parallelism with the reference edge of the glass plate. Due to the relatively large size of the quartz masses this is about as accurate an orientation as can be achieved with Polaroid as an analyzer, or the use of supplementary biquartz plates,[20] or through the use of a photoelectric cell.[21] A third piece of Polaroid with marked direction of the plane of polarization may be used to calibrate the reference edge of the stage.

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[17] A good example of the complexities introduced in the processing of quartz plates due to the lack of fundamental crystallographic knowledge is seen in some of the elaborate methods developed for determining the electric axes. One method involved cutting a ring perpendicular to the optic axis and applying an alternating electric field in an evacuated chamber and noting the direction of the X-axes from the luminous phenomena (Geibe, Erich and Scheibe, Adolf, Method for determining electric axes in crystals: *U. S. Patent Office* No. 1,720,659, July 16, 1929). Another method involved cutting a Z-section and determining the piezoelectric effect at different parts of the surface by a probing electrode and plotting the results (Dawson, Leo H., Method and apparatus for determining the direction of the electric axes of crystal quartz: *U. S. Patent Office* No. 1,866,454, July 5, 1932).

[18] Rivlin, R. S., Grinding and scratching crystalline surfaces: *Nature*, **146,** 806–807 (1940). The figure is pictured by Willard, G. W., *Bell Syst. Tech. Jour.*, **23,** 36 (1944).

[19] Mounting and angular-view stauroscopes may be obtained from The Polarizing Instrument Co., Inc., 41 E. 42nd St., N.Y.C.

[20] Made from basal sections of right- and left-quartz 4 mm. thick. A piece of transparent Scotch tape may be used as a substitute by cutting a square piece diagonally, turning one half upside down and joining again on the diagonal line. It may be placed on a glass slide below the analyzer or directly on a glass mounted Polaroid. Both halves of the tape will show the same color when the quartz is at extinction, and different colors when not

Angular-View Stauroscope. This instrument (Fig. 11) is essential for finding the electric axis direction in wafers to guide in dicing them, and

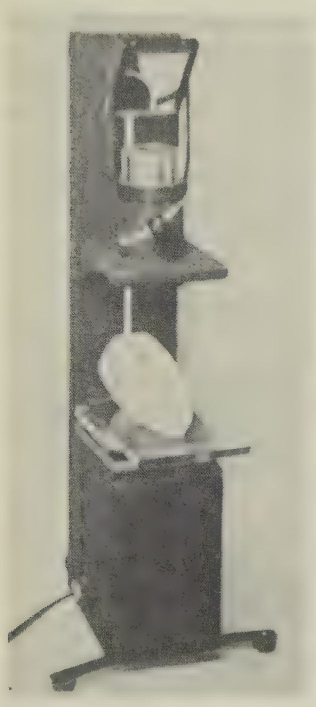

FIG. 10

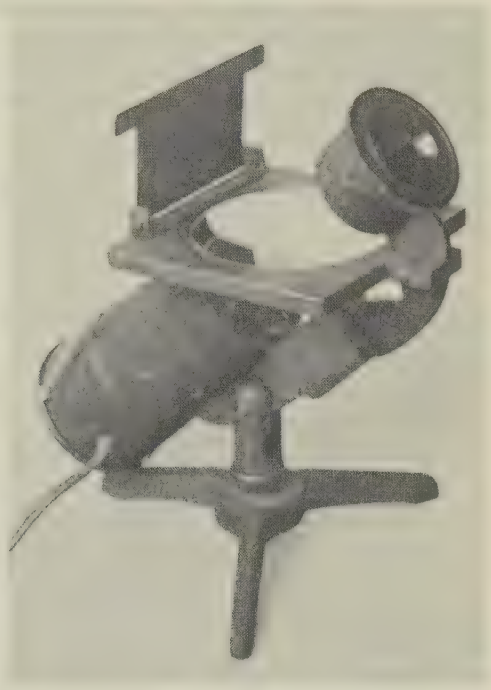

FIG. 11

FIG. 10. Mounting stauroscope used for setting Z (*c*) of crystal, X-section or bar parallel to long reference edge of glass mounting plate. Instrument is simply a pair of crossed Polaroids set with standards so that plane of polarization of one Polaroid is parallel or perpendicular to reference edge on stage.

FIG. 11. Angular-view stauroscope. Stage set at approximately 45° to tube carrying crossed Polaroids. Only one complete extinction position is obtained for AT- and BT-wafers. The marking guide is used to rule this direction left to right, and an arrow head marked away from the observer to indicate optic axis direction which in this position is approximately perpendicular to tube.

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quite at extinction. Split-field Polaroids with the plane of polarization of each half 3° apart are also available.

[21] Since large amounts of light are available, a photovoltaic type of cell such as the Weston Photronic cell may be used without the necessity of adding an amplifier. See Whitford, A. E., Chap. X, in Strong, John, *Procedures in Experimental Physics*, Prentice-Hall, Inc., New York (**1938**).

for finding the electric and optic axis directions in wafers and blanks preparatory to rechecking them by x-ray measurements. For the latter purpose, the instrument permits setting the wafer or blank in the crystal holder in the proper position for x-ray measurement. It differs from the conventional stauroscope in that the stage is at an angle of about 45° to the axis of the tubes carrying the crossed Polaroids. The field of the polarizer is made small. The instrument is usually equipped with a rule to mark the direction of the electric axis (left to right in the extinction position), and an arrow pointing away from the observer to indicate the optic axis direction.

If a BT-wafer is revolved on the stage it will dim out twice and extinguish completely only in one position; in this position the optic axis direction is perpendicular to the axis of the tube and the electric axis is left to right. When the section is rotated 180° from the extinction position, the line of sight will be parallel to the optic axis as evinced by the characteristic color bands due to rotatory polarization.

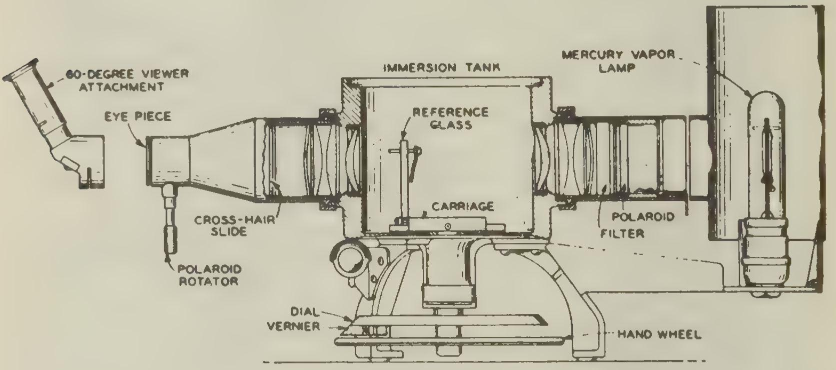

FIG. 12. Conoscope designed at Bell Telephone Laboratories. The instrument is invaluable for rapidly and accurately locating the optic axis of faced or defaced quartz and may also be used to determine hand. (Drawing from W. L. Bond, *Bell Syst. Tech. Jour.* **22,** 224, 1943.)

Conoscope.[22] The conoscope is a device which has been in use by crystallographers for over a century for examining minerals in convergent light and measuring their optic axial angles. For the purpose of the quartz oscillator industry, the instrument has been greatly improved[23] over the crude Fuess model which is occasionally found gathering dust in the older mineralogical laboratories. The optical system has been carefully designed, and the analyzer can be rotated to determine the hand of the quartz (Fig. 12). The trough has a rotating stage on the bottom with a track for a jig or a vertical glass reference plate to carry sections. A graduated circle and vernier permits readings to 6′. The trough is large enough to hold a mass of quartz weighing about one kilogram. The light source is a mercury vapor lamp whose rays are converged by the optical system to enter the quartz at a cone angle of 40°. It is essential that the quartz be immersed in a liquid with an index of refraction of 1.544, and a number of such liquids are listed by Bond. Many of the liquids used have one or more of the objectionable features of being viscous, nasty smelling, skin irritating, or becoming so dark from their dry cleaning action on the quartz and air as to become opaque. Lindol[24] (ortho-free tricresyl phosphate) has an index of 1.555 (at 20°C) and may be lowered to 1.544 by mixing with a miscible liquid of lower index; it seems to have the only fault of being viscous.

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[22] Manufactured by Bulova Watch Co., N.Y.C. and Shuron Optical Co., Geneva, N. Y.

[23] Bond, W. L., *op. cit.*

Quartz crystals, faced or defaced, and sections may be quickly oriented by centering the optic axis figure upon the cross-hairs (one horizontal and three vertical) in the eyepiece. The image is an interference figure due to axial birefringence[25] resulting in rotation of the plane of polarization. Rays differing in convergence pass through different thicknesses of quartz and each colored ring represents those rays which emerge at the surface of the quartz vibrating parallel to the plane of polarization of the analyzer. The color of the center of the first circle, is the complement of that absorbed by the interference of the two circularly polarized rays travelling in the direction of the optic axis. The color of the rings is due to interference of elliptically polarized rays; only at the farthest rings where isogyres appear are the effects of interference of plane polarized rays, ϵ and ω to be seen. Although optical twinning confuses the pattern, the rings remain clear enough for orientation.

Z-Sections in the Conoscope. The first use that seems to have been made of the conoscope was the testing of the trueness of a plane cut perpendicular to the optic axis. The stage was set a 0° with a vertical glass plate in the tank normal to the beam of light. The section to be tested was held against the glass reference plate and the position of the optic figure was observed. If the saw cut was true, the rings would center about the cross-hairs (Fig. 13). Any divergence would be apparent and could be measured by rotating the Z-section upon the glass plate with the stage set at 0°, until the center of the rings had travelled to the maximum distance from the cross-hairs, and then reading the circle. Since two planes intersect in a straight line, it is obvious that in the position of maximum divergence of Z, the intersection of the true (0001) plane (the desired cut) and the plane which has been cut (resting against the glass reference plate) is now vertical, and that this vertical line is one of zero deviation. The maximum divergence is at right angles to this line and in the direction of the optic figure seen in the conoscope. This direction is marked on the section, which can be taken to a lapping wheel and lapped on the "high side"; jigs have been developed for the purpose. The section is rechecked until no divergence is apparent.

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[24] Sold by Celluloid Corp., 180 Madison Ave., N.Y.C. Specify ortho-free for the ortho-form is toxic.

[25] On the axial birefringence of quartz, see Sosman, Robert B., *op. cit.*, pp. 667–669.

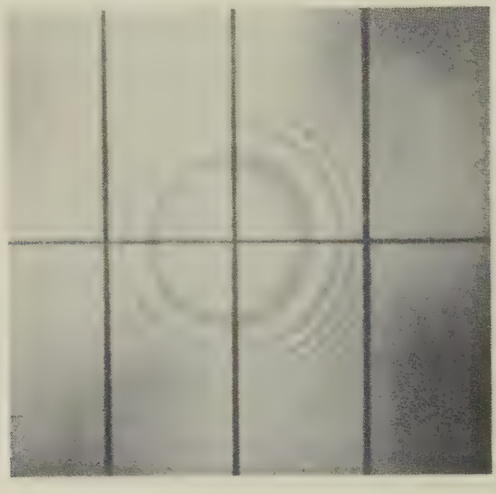

FIG. 13. Series of rings centered on cross-hairs of conscope show optic axis is parallel to reference track in immersion tank.

Mounting Defaced Quartz in the Conoscope. Because the optic axis direction may be so readily found in the conoscope, and so accurately centered, the instrument has been invaluable in cutting defaced quartz. It permits cutting at least one true plane, perpendicular to the optic axis, which is easily accomplished, after which the quartz can be cut as faced quartz.

For mounting purposes, a ground surface of about one square inch parallel to the optic axis is desirable. The quartz is turned in the oil in the conoscope until the optic axis is centered; is carefully lifted out, and without losing the optic axis direction, is pressed down on a large iron lap carrying wet #100 silicon carbide. The surface is wiped clean, and the quartz is set in the bottom of the conoscope to make sure that the mounting surface is not more than about 10° from parallelism with Z. The optic axis direction is marked by a line drawn on the ground surface cemented to a glass plate with Z approximately parallel to the reference edge using the mounting stauroscope. The mount is locked to a simple transfer jig, which fits on the track in the conoscope Screw adjustments on the jig permit tilting the mount until the optic axis figure is on the horizontal cross-hair. Divergence from the vertical cross-hair may be read on the graduated circle and marked on the plate for correction at the saw.

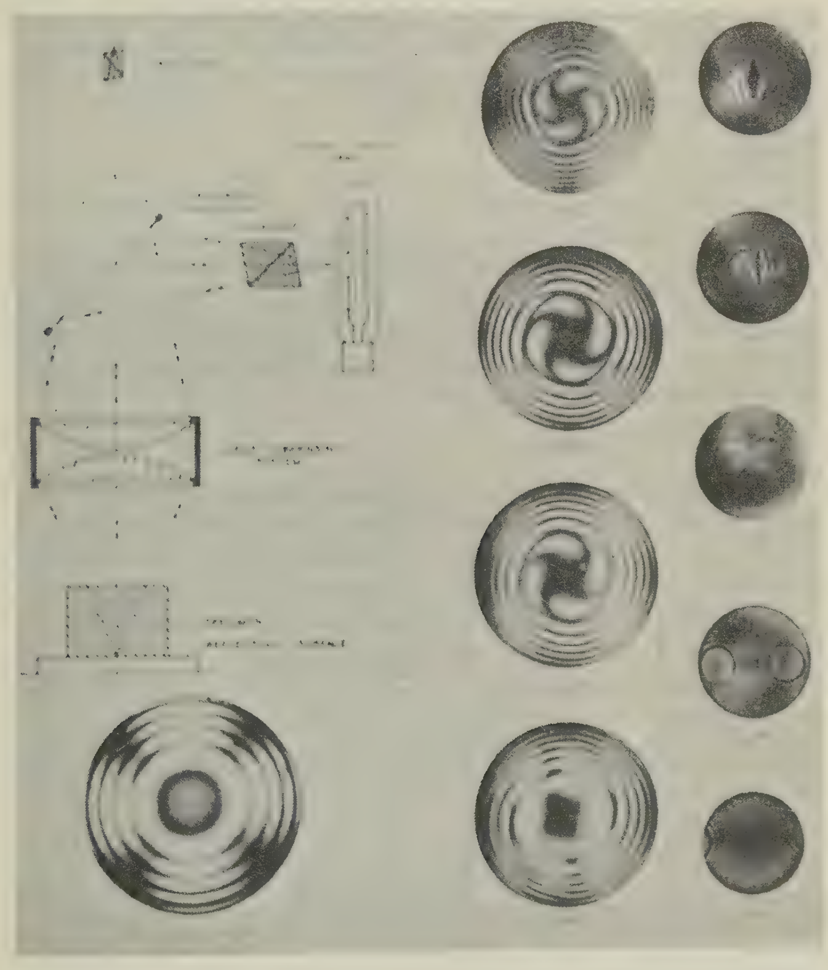

FIG. 14. Airy-spiral conoscope. A novel instrument developed in England for testing trueness of Z-sections. On single light transmission the figure (lower left) is similar to that seen in conoscope. On passing light back through the section by means of a mirror, the Airy-spiral figure is observed. The latter is sensitive to slight inclinations of the optic axis as indicated by the series of figures for various inclinations. (From C. F. Booth, *Jour. Inst. Elect. Eng.*, **88**, 97, 1941.)

Airy-Spiral Conoscope. This conoscope, of novel design, was developed in England for testing the trueness of a Z-section.[26] The Z-section is placed upon a mirror which can be revolved (Fig. 14). Light from a monochromatic source such as a sodium vapor lamp is polarized, reflected by a thin glass plate through condensing lenses to the Z-section. The mirror reflects the light back through the quartz to the analyzer and the image seen is like that of the well known Airy's spirals formed by superimposing a basal section of right-quartz upon a basal section of left-quartz in convergent polarized light. The figure is very sensitive to errors of angle in cutting the Z-section which are detected by the divergence of the two images.

Projection Conoscope. If a Z-section of quartz, a few mm. thick is sandwiched between a pair of crossed Polaroid films, and held close to the eye, the optic axis figure due to rotation of the plane of polarization can be seen. By holding a point source of light (such as a projection or automobile light bulb) close to the sandwich, it is possible to project the image to a not too distant screen. It is not difficult to rig up a device to project the optic axis image of Z-sections upon a screen which can be ruled to indicate divergence from trueness.

ORIENTATION OF ETCHED QUARTZ

The etching processes are extremely important in the manufacture of quartz oscillator-plates. The pin-hole light figures are used for *orientation* of blocks, bars and sections. Raw crystals, blocks, and wafers are etched to reveal *twinning*.[27] In the *final finishing* operations, the blanks are etched to frequency; this is an ideal finishing procedure[28] since it results in a stable surface which does not "age."

Etching Conditions. Hydrofluoric acid was used almost exclusively in the quartz industry up to a few years ago but its use caused serious industrial hazards and inconveniences. Today nearly all manufacturers use a concentrated water solution of ammonium bifluoride. The elaborate precautions required in the use of HF are not necessary in using the bifluoride although certain safety measures should be followed in using all fluoride reagents. At ordinary room temperatures, 33 gms. in 100 cc. of water is practically the limit of solubility. The reaction is strongly endothermic and solution is promoted by warming the water. HF etches quartz two to three times faster than ammonium bifluoride. Although their pin-hole light figures differ in some details the general description given here applies to figures obtained with both solvents. In general HF produces larger and coarser etch pits and the bifluoride forms a larger number of finer etch pits. Many other solvents have been used to etch quartz such as potassium carbonate, sodium fluoride, etc.

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[26] Booth, C. F., *op. cit.*

[27] Gordon, Samuel G., The inspection and grading of quartz: *Am. Mineral.*, this issue.

[28] Frondel, Clifford, Etching techniques in the manufacture of quartz oscillator plates: *The Radio Eng. Digest:* **1**, 31–36 (1944); Final frequency adjustment of quartz oscillator-plates: *Am. Mineral.*, this issue.

Natural crystal faces are very slowly etched but roughing the surfaces by lapping or sand blasting prior to etching decreases etching time and produces more evenly distributed and better developed etch figures. The coarser the abrasive used the shorter is the etching time. In etching-to-frequency in the final finishing operation where only 0.00004″ to 0.00005″ is removed for final frequency adjustment and to effectively prevent ageing, the machine lapping finish must be carefully controlled to obtain predictable and reproducible results. Crystals should be cleaned in a solvent such as carbon tetrachloride or trichlorethylene ("Tromex") before etching. When holes, crevices and blue needles are present, the quartz is rapidly etched to a fibrous aggregate.

The etching rate increases markedly with temperature. Oscillator plates with #303 optical flour machine lap finish decreased approximately 0.00056″ in thickness when etched two hours in a 30% solution of ammonium bifluoride at 75°C. compared to approximately 0.00014″ being etched off under the same conditions at 25°C.[29] Large masses of quartz such as raw crystals, X-blocks, Z-sections, etc., must be etched at or near room temperatures to prevent cracking due to differential thermal expansion. At least ten to twenty hours etch is required to produce a surface for orientation and inspection purposes. Rough-cut wafers obtain an etch sufficient for twinning inspection in 1½ hours at 50°–60°C. in a 30% ammonium bifluoride solution. Agitation of the etch solution by rocking the container or constant stirring also speeds the action. The thickness decrease of crystals etched under the conditions stated above at 75°C. but with the etch solution constantly stirred is approximately 0.00071″.

The tendency in the crystal industry has been to stop etching as soon as the matt surface appears. The first effect of etching is to dissolve the thin submicroscopic surface layer which has been shattered by sawing or lapping and better results are obtained if the etching is continued into the solid quartz.

Etch Figures on Quartz. Solution is much more rapid (by a factor of over 100 times) parallel than perpendicular to the optic axis.[30] The minor rhombohedral (z) faces are more rapidly etched than the major rhombohedral (r) faces; the latter give a coarser and more geometrical pattern (Fig. 19). Kalb[31] and Ichikawa[32] have studied natural etch figures. There have been several classical studies on the etching of spheres,[33] crystals and sections.[34]

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[29] These thicknesses were computed from frequency measurements using the formula: Thickness (inches) = K/Frequency (kilocycles) where $K = 100$. ½″ square BT-cut crystals, 7.8 MC starting frequency.

[30] Mügge, O., *Rosenbusch Festschrift*, 96–126, Stuttgart (1906).

[31] Kalb, George, Beitrage zur Kristallmorphologie des Quarzes. I–V: *Zeit. f. Krist.*, **86**, 439–452 (1933); **86**, 453–465 (1933); **90**, 163–185 (1935); also **86**, 1–7 (1933); **89**, 400–409 (1934); also literature references contained therein.

[32] Ichikawa, S., Studies on the etched figures of Japanese quartz: *Am. Jour. Sci.*, **39**, 455–473 (1915).

[33] Meyer, O., and Penfield, S. L., Results obtained by etching a sphere and crystals of quartz with hydrofluoric acid: *Trans. Conn. Acad.*, **8**, 158–165 (1889). (Note remarks of Van Dyke, p. 403 of article cited below, regarding hand of crystal used.) Gill, A. C., Beiträge zur Kenntniss des Quarzes: *Zeit. Kryst.*, **22**, 97–128 (1893). Mugge, O., *op. cit.* Nacken, R., Atzversuche an Kugeln aus Quarz und α-Quarz: *Neues Jahrb. Mineral.*, **1**, 71–82 (1916). (Also describes etching of spheres in sodium metaphosphate at 700°C. to determine symmetry of high temperature quartz). Bond, W. L., Etch figures of quartz: *Zeit.*

Photomicrographs of typical etch figures on various planes of quartz and their orientation are shown in Fig. 15. Sections (*a*) to (*e*) show etch figures on both sides of an electrical twin boundary indicated by pairs of lines on the rim of the photograph. These sections were cut 0.095″ thick and lapped successively in #320 and #600 SiC and finally 0.002″ lapped off with #303 Al_2O_3. The sections were thoroughly cleaned and etched 4 hours with 30% NH_4HF_2 at 50°C. Section (*f*) is an untwinned section lapped only with #600 SiC and etched 2 hours under the same conditions. (*a*), (*b*) and (*c*) were cut at 35°15′ (AT), 38°13′ (major-minor rhomb plane) and 49°30′ (BT), respectively, to Z which is indicated by the arrow and is inclined to the plane of the paper. The characteristic "shingle" structure is on the side more nearly parallel to the major rhomb *r*, the "ripple" structure on the side nearly parallel to the minor rhomb *z*. The characteristic parallelogram light figure described below is seen as being due to parallelogram-like etch figures (*d*) on that side of the X-plane with −X up. The indistinct types of etch figures on the +X side of the X-plane produce the arrow-like light figures. The Y-plane shows etch figures (*e*) pointing in opposite directions along Z on either side of the electrical twin boundary. The characteristic triangular light-figure patterns on etched Z-sections is seen to be due to distinct and geometrical etch figures which appear like sections of rhombohedra standing on a corner forming a triangular figure in horizontal sections. The edges between the rhomb faces appear to be parallel to the Y-axes. In an electrical twin these figures are rotated 180° about Z.

Orientation of Quartz by Etching. The characteristic symmetry expressed in well developed quartz crystals by the disposition of the zone of minor faces *x* $(51\bar{6}1)$, *s* $(11\bar{2}1)$ and *z* $(01\bar{1}1)$ above the negative (on compression) end of the polar X-axes is shown also by the symmetry of the etch figures on natural crystals and those produced by solvents on sections of quartz. Diagnostic also are the pits etched on the *r* and *z* faces and since these planes become coplanar in twinned crystals it is a

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Krist., **99**, 488–498 (1938). (Gives photomicrographs of etch figures on a hollow quartz sphere etched with HF.) Van Dyke, Karl S., On the right- and left-handedness of quartz and its relation to elastic and other properties: *Proc. Inst. Radio Eng.*, **28**, 399–406 (1940).

[34] Descloizeaux, A., Memoire sur la cristallisation et la structure interieure du quartz: *Ann. Chim. et Phys.* (3) **45**, 129–316 (1855); *Memoires Acad. Sci.*, (Inst. Imp. France) **XV**, 404–614 (1858). Leydolt, Franz, Über eine neue Methode, die Struktur und Zusammensetzung der Krystalle zu untersuchen, mit besonderer Berücksichtigung der Varietaten des rhomboedrischen Quarzes: *Sitzber. d. kais. Akad. d. Wiss. Wien* (*Math. Natur. Classe*) **XV** (**1**), 59–81 (1855). Molengraaff, G. A. F., Studiem über Quarz: *Zeit. Kryst.*, **14**, 173–201, 1888; **17**, 137–176 (1889). Bomer, A., Beitrage zur Kenntniss des Quarzes: *Neues Jahrb. Mineral. B. B.*, **7**, 516–555 (1891). Honess, Arthur P., *The Nature, Origin and Interpretation of the Etch Figures on Crystals*, John Wiley & Sons, New York (1927). Booth, C. F., *op. cit.*

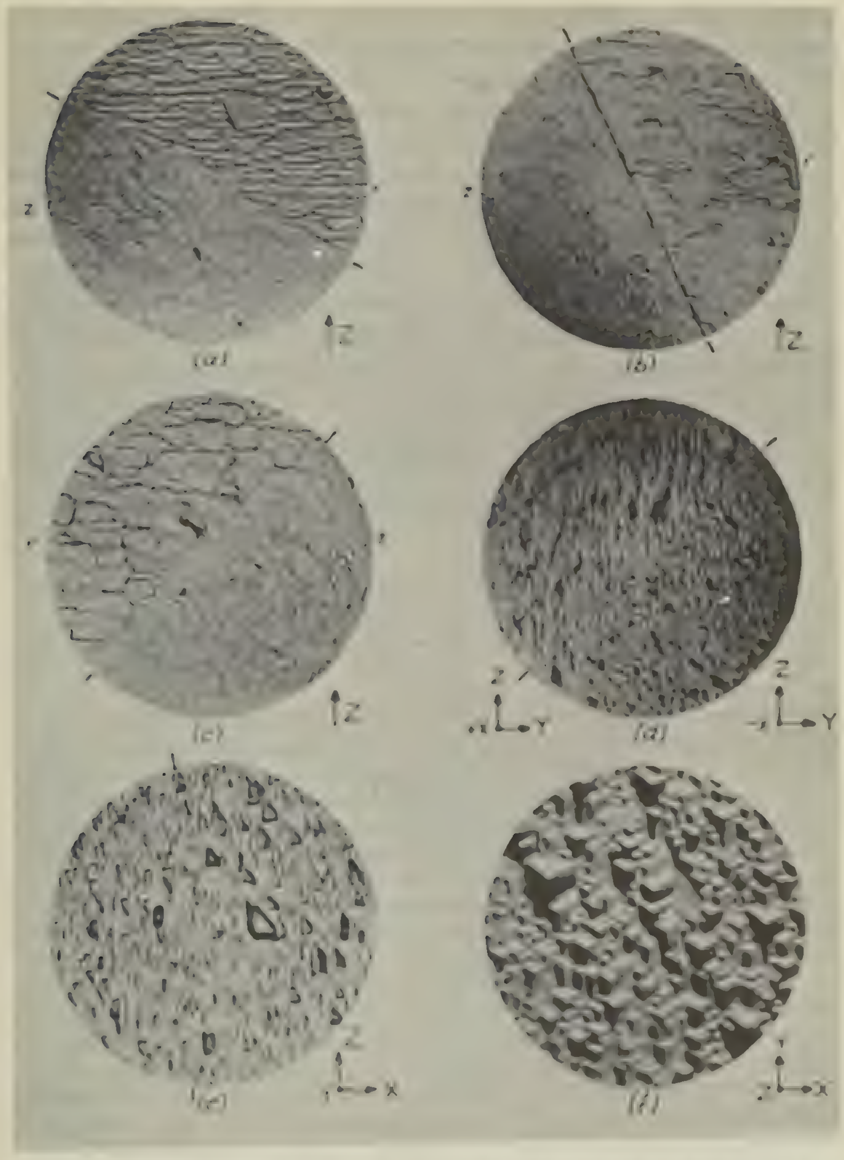

FIG. 15. Photomicrographs of etch figures on quartz produced on finely lapped surfaces etched with NH_4HF_2 (see text). Electrical twin boundaries in (a) to (e) indicated by pairs of lines on periphery of each photograph. (a) AT-plane, ZZ′ = 35°15′, magnification 60×; (b) Major-minor rhomb plane, ZZ′ = 38°13′, 60×; (c) BT-plane, ZZ′ = 49°30′, 60×; (d) X-plane, 110×; (e) Y-plane, 110×, (f) Z-plane, 175×.

method of distinguishing them. The etch pits, or their refracted and reflected light pattern figures, are the sole infallible and unambiguous criterion for the orientation of quartz.

The Abbe C. Gaudefroy[35] in 1931 called attention to the photographs of DeGramont and Mabboux[36] and the use of light figures seen on reflecting a distant light source (several meters away) from HF etched basal sections of quartz for fixing the position of the 2-fold and 3-fold axes to about one degree, the sense of the piezoelectric axes, and the hand of the crystal even in defaced quartz. In 1933, Gaudefroy described in detail the now familiar parallelogram and arrowlike light figures which he called *motif asterique*, and which he obtained by reflection from etched spheres of quartz.[37]

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[35] *Bull. soc. franc. min.*, **54**, 80 and 108 (1931).

[36] "Supplement a la notice sur les travaux scientifiques de M. Armond de Gramont, *cit.* Paris, 1929."

[37] Gaudefroy, C., Sur les groupements de cristaux de quartz à axes parallèles: *Bull. soc. franc. min.*, **56**, 5–63 (1933). Light figures of etched spheres of quartz were used by Nacken, *op. cit.*, for showing the symmetry of quartz.

The posthumous work of Armond De Gramont, *Recherches sur le quartz piezoelectrique*, Ed. de la Rev. d'Optique Theorique et Exp., Paris (1935), seems to have been the inspiration for some of the orientation devices in use. Several devices and methods have been recently described and some have been assigned patents for use in orienting etched sections and crystals by means of their light figures:

Hawk, Henry W. N., Art of examining quartz: *U. S. Patent Off.* No. 2,264,380, Dec. 2, 1941. One side of a *Z*-section is etched and the other remains unetched or is polished. Unlike most of the other methods of using the light figures, the etched side is toward a pin-hole source of light and the figure is observed from the unetched side of the image or is projected on a screen. The method claims to determine the *X*-axes which are parallel to the sides of the observed equilateral triangle, their polarity, and direction of the major rhombohedral faces from the position of the triangle.

ANON., *Manual for the construction and use of the rodometer*, Scott Lab., Wesleyan Univ., Middletown, Conn., April 1943. This paper describes more elaborate equipment in which the light figure is projected on a ruled screen by means of a photographic lens. The unetched end of the crystal is immersed in oil to reduce refraction and reflection. The great difficulty in applying such methods to actual production is that in order to transfer the orientation to a saw, it is necessary to employ gimbals and the like to hold the crystal for cementing and processing. See for example Waesche, Hugh H. and Wolfskill, John M., A method of orientation and sawing small unfaced quartz: Inf. Bull. No. 5, *Handbook for the Manufacture of Quartz Oscillator-Plates*, Off. Chief Sig. Off., War Dept., Washington, D. C., March 1943.

Several devices are described by Willard, G. W., Use of the etch technique for determining orientation and twinning in quartz crystals: *Bell Syst. Tech. Jour.*, **23**, 11–51 (1944). These include a *reflection oriascope* (from orientation- and scope) for detecting the sense and approximate direction of the electric axes. The rays of light are brought to focus by a pair of condensing lenses on the surface of an etched *Z*-section and reflected back upon a viewing screen. The trigonal figure is shifted about 12° clockwise or counterclockwise from the *X*-axes depending upon the hand of the crystal and ruled with a preset marking

Production of Light Figures on Etched Sections. The characteristic light figures are the result of reflecting and refracting light rays from the facets of the etch pits or hills. The figure obtained is that of the upper etched surface of the section. If the lower surface in contact with the pin-hole disturbs the effect, it may be immersed in a layer of oil in a glass vessel placed upon the pin-hole. The image is a composite of several images and is not in one plane. In photographing it, it is necessary to focus on a plane about half way in the section. A point source of light is used such as a distant light, an illuminated pin-hole, or a light brought to a focal point by a condensing lens. One of the best methods of observing the figure is to place the section on the stage of an ordinary microscope, remove the entire microscope tube and illuminate the section by means of the condenser (Fig. 16). The most commonly used device is a box containing an electric light bulb which illuminates a tiny pin-hole (made with a #60 or smaller drill) in a sheet of metal. Sometimes several pin-holes are used to obtain greater accuracy in ruling the axial directions; each pin-hole produces a separate figure. A hinged ruler is convenient for marking directions of the axes.

Light Figures on Etched Z-Sections. The figures obtained upon etching basal sections of quartz vary markedly with the preparation of the surface and etching time. The figures shown in Fig. 17 were obtained on a

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template. In the *pin-hole oriascope*, light is passed through a pair of condensing lenses to a mirror which reflects it to a diffusing screen in contact with the pin-hole. Preset marking guides are provided for ruling axial directions on Z- or X-sections.

In the *reflection twinoriascope*, used for detecting usable portions of electrically twinned AT, BT, CT and DT wafers, the mounting table is tilted to an angle depending upon the angle of cut of the wafer being examined. Deeply etched wafers also show a characteristic light reflection figure when examined in a beam of light brought to focus by a condensing lens. These figures distinguish parts of electrically twinned wafers as being cut respectively at negative or positive angles to Z: those on the negative angle side have the appearance of a golf club (usable side in case of BT- and DT-wafers); the positive angle side shows the silhouette of a spoon (usable side for AT- and CT-wafers).

Gerber, P. D., Piezoelectric crystal: *U. S. Patent Off.*, No. 2,218,489, Oct. 15, 1940; Apparatus for examining quartz: *ibid.*, No. 2,313,143, March 9, 1943. The first patent describes a method for locating and determining the polarity of X-axes and the hand (the Herschel convention is used) of etched Z-sections and the second patent describes a device for this purpose. The etched Z-section is placed on a rotating stage and light from a projector at 30° to the surface is observed at an angle of 10° to 15° from the horizontal surface. It is stated that the sharpest reflections for this set-up are obtained 10° to 15° clockwise or counterclockwise from $-X$, depending upon the hand of the crystal (note similarity to the above description). A ruling guide is provided for marking the specimen.

Eckert, J. F., Art of examining quartz: *U. S. Patent Off.* No. 2,328,968, Sept. 7, 1943. This patent relates to the use of a parallelogram light figure observed on a pin-hole light box but contains a serious error. The long side of the parallelogram is stated to be the direction of Z, and the short side r. These, of course, are actually just in reverse.

surface cut with a 12″ Norton metal bonded diamond blade (but not lapped) and etched 28 hours in 30% ammonium bifluoride solution at 68°F. Z-sections prepared in this way show two characteristic sets of three spots. By drawing connecting lines between the spots of either set,

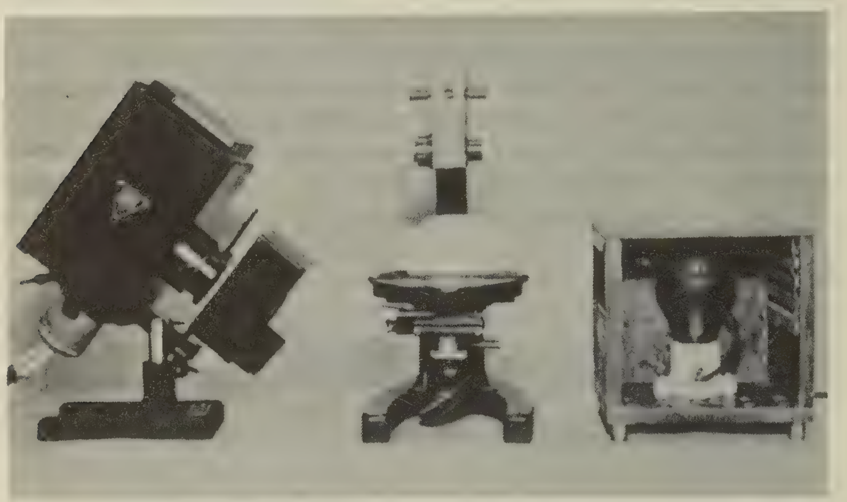

FIG. 16. Light figures of etched sections may be observed by placing section on an illuminated pin-hole. A microscope stage with tube removed and condenser in place (left) or a box with a 60-W light bulb and a #60 drill hole (right) may be used.

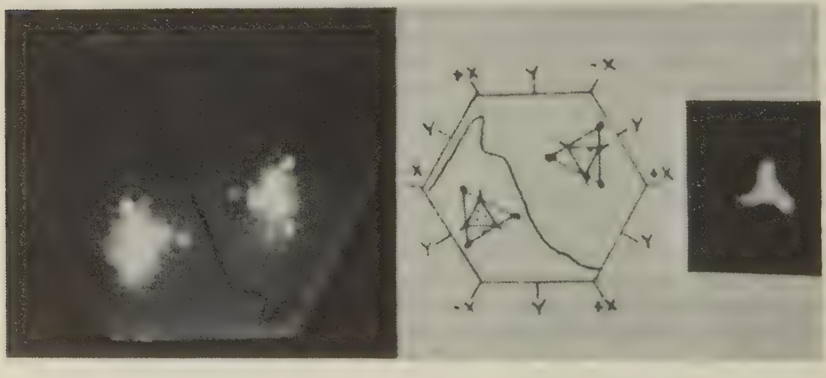

FIG. 17. Light figures on electrically twinned Z-sections showing three typical equilateral triangles formed by three sets of spots useful for orientation purposes. The outer set of brightest spots form equilateral triangle with sides parallel to Y-axes. The fainter spots with tails form a triangle with sides parallel to X-axes, The propeller-like figure becomes more distinct with long etching and shows hand of crystal (both right-handed in this case).

they form a perfect equilateral triangle when the section is perpendicular to the optic axis. A faint group of three spots with tails are at the corners of a small equilateral triangle whose sides are parallel to the X-axes. The tails of the spots are in the direction of the Y-axes. A larger equilateral triangle is formed by three very much brighter spots and its sides are parallel to the Y-axes. In electrical twinning the small triangles are related as by a mirror image and the large triangles are reversed 180°. Other details appear as shown in the photograph but these have not yet been analyzed.

With a longer etch, both sets of dots practically disappear and the trigonal trapezohedral symmetry becomes more obvious. The figures assume the shape of three-leaved propellers which show the hand of the crystal (right-hand screws for right quartz, etc.), the arms roughly indicating the direction $-X$. These propellers are also visible but less apparent in sections with a shorter etch and are most distinct on sections which have been lapped prior to long etching. When prepared in the latter way, a third set of dots become apparent in the center of the propeller (which becomes more distinct) and forms a tiny equilateral triangle. The sides of this triangle are rotated approximately $13° \pm 2°$ from the Y-axes. These two triangles are related by a 180° revolution in electrical twinning.

The orientation of the light figures is used to determine the approximate direction of Y, which is ruled on the section. An accurate determination may be made by *x*-ray reflection from the side of the Z-section using the $(11\bar{2}0)$ or $(10\bar{1}0)$ atomic planes and the right angle marking guide as described in the following section.

The direction of the electrical axes is precisely indicated by the twin boundaries of optical twinning if such twinning is present. Electrical twinning is identifiable as irregularly bounded areas.

Orientation of Etched X-Sections and Y-Bars. The "parallelogram" light figure obtained on viewing the negative (on compression) side of an etched X-section laid on an illuminated pin-hole, has proven to be a most useful aid in the precise orientation of quartz (Fig. 18). It indicates the trend of a zone of minor faces *x* $(51\bar{6}1)$, *s* $(11\bar{2}1)$ and *z* $(01\bar{1}1)$, were these on the crystal. The long, fainter sides of the parallelogram mark the slope of the major rhomb faces; i.e., they are parallel to the intersection edge of *r* $(10\bar{1}1)$ and the plane of the section $(11\bar{2}0)$. The shorter brighter sides are parallel to the optic axis direction, (Z, *c*). The positive side of the X-section shows a figure often referred to as the "*Z*" or arrow-figure. The horizontal line indicates the Y-axis and the two appendages at either end show the direction of the minor rhomb faces.

Since AT, CT, ET and GT cuts are not far from parallelism with the minor rhomb face *z*, Fig. 1, while the other standard cuts, BT, DT and

FT are not far from parallelism with the major rhomb, *r*, and since the trace of the rhomb plane as well as the optic axis direction are so clearly defined by the parallelogram light figure, an unambiguous and infallible guide is furnished for marking the directions of the cuts, without the bother of determining the hand of the quartz in a polariscope or the polarity of an electric axis in the piezometer. A simple rule, useful in directing the cutting of X-sections is given in the procedure of the accompanying paper by the writers on Cutting Schemes.

At the same time, examination of the etched X-section will reveal electrical twinned areas, which may be sawed apart, if of sufficient size to yield blanks since electrical twins are composed of reversed individuals, and therefore one portion of a wafer cut across both would be a miscut on the wrong side of the optic axis. Once the crystal has been cut at an angle to the optic axis, electrically twinned sections cannot be salvaged.

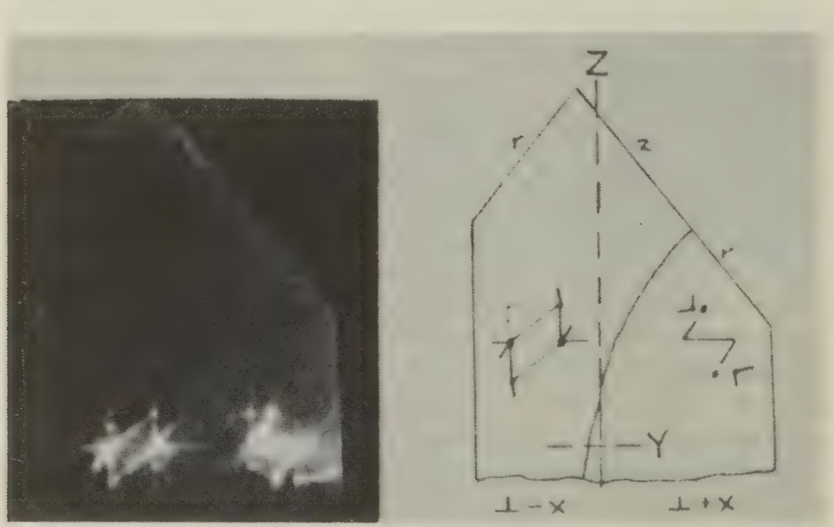

FIG. 18. Light figures on electrically twinned X-section showing characteristic parallelogram light figure when viewed along −X and "*Z*" figure viewed along +X. The use of these figures eliminates the necessity for hand or polarity determinations and are an infallible guide for orientation purposes.

Orientation of Etched Wafers. Wafers are etched for two purposes: to identify and permit the marking out of optical twinned areas and to identify the useful areas of electrical twins. In electrical (180°) twinning, the *r* and *z* faces are coplanar so that one side of a twin boundary on a wafer is usable, while the other side will be equivalent to a miscut of twice the angle of the cut. Pertinent data taken from Willard for correct and miscuts are given in Table 1.

TABLE 1[a]

Cut	ZZ′	Frequency Constant (Freq. (KC)× Thick. (mm.))	Temperature Coefficient (Parts/10^6/C.°)
AT	+35°	1670	0
	−35°	2400	+30
CT	+38°	3080	0
	−38°	2100	−30
BT	−49°	2560	0
	+49°	1880	−55
DT	−52°	2060	0
	+52°	2850	+45

[a] Data from G. W. Willard, *op. cit.*, p. 48.

As was pointed out above, AT- and CT-cuts are near parallelism with *z* while BT-cuts are near *r*. Since *r* and *z* develop different patterns when they are etched, it is a simple matter to distinguish the two planes by

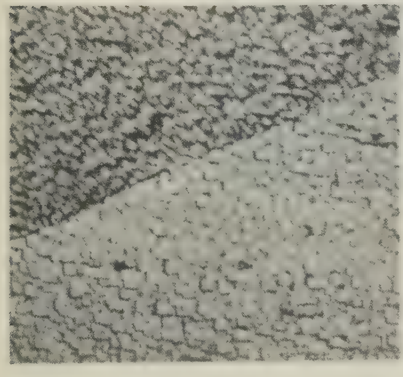

FIG. 19

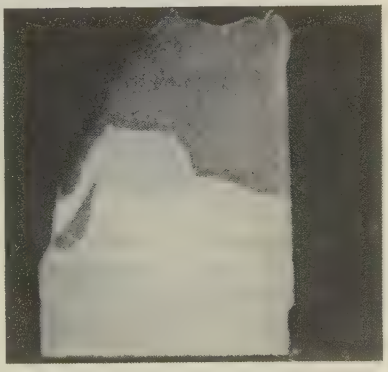

FIG. 20

FIG. 19. The useful portion of a well-etched electrically twinned wafer may be determined by its translucency. The useful side for BT-cut wafers, below, is nearly parallel to *r* has the "shingle" structure, is more translucent than that parallel to *z* which has the "ripple" structure and is the useful side on AT-cut wafers. This wafer was etched very deeply in HF, one surface polished, placed in a slide projector and the image focussed on a screen which was photographed.

FIG. 20. The difference of reflectance from well-etched electrically twinned wafers is applied in determining the useful portion. When examined in a twinoscope the useful side of BT-cut wafers reflects brilliantly once in a 360° revolution when viewed at about 22° from the incident light beam.

their etch figures, either in misoriented crystals, or in coplanar electrical twins.

If a well-etched electrically twinned wafer is examined under a binocular microscope, or Scotch-taped to a glass slide and projected on a screen, the characteristic patterns of the two planes will be apparent. Etched major rhomb faces (r $10\bar{1}1$) show striking rhombohedral facets, which may be described as a *shingle* structure (Fig. 19); the minor rhomb faces (z $01\bar{1}1$) have a less definable pattern which may be designated a *ripple* structure. The AT- and CT-types of plates must be diced from areas showing the ripple structure, while BT-plates must be diced from areas showing a shingle structure, provided, of course, that the wafers have been *cut at the proper angle.* If the latter condition has been followed, only one side of the twin boundary is usable, and the other side is worthless.

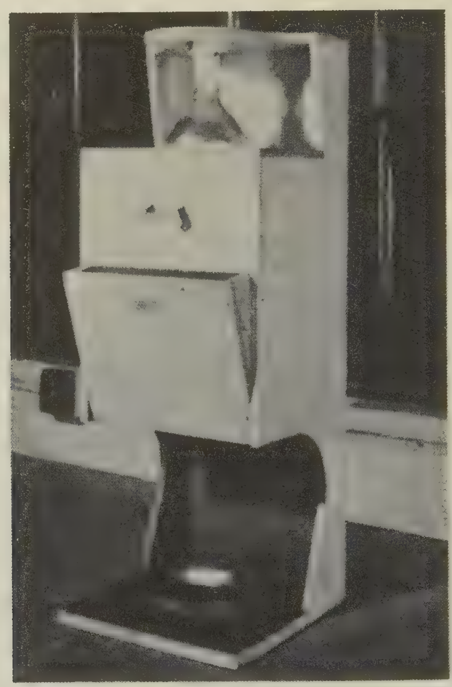

FIG. 21. Twinoscope. Consists simply of a spot-light, turntable and viewing slot approximately 22° from the normal for determining useful portions of electrically twinned wafers by differences in light reflectance.

In actual practice, wafers are examined in a spot-light set at an effective angle, or a *twinoscope* (Fig. 21) which merely consists of a vertical case containing a light which can be adjusted to strike a wafer on a stage (which can be revolved and is painted black) at an angle to cause reflection from the etch figures of the wafer. The useful side of AT-wafers has a frosty, more opaque appearance and the useless side is more translucent and has a brilliant, almost metallic luster. When BT-wafers are revolved on a stage in the spot-light beam, the useful side will reflect strongly *once* (Fig. 20) in a 360° revolution, provided the position of the spot-light has been properly preset by means of a standard. The reflecting r $(10\bar{1}1)$ planes produced on etching are at about 11° to the normal of a BT wafer. The usual routine is to roughly pencil the twin boundary, and to scribble over the flawed areas, including any parts showing optical twinning.

PRECISE ANGULAR CONTROL OF QUARTZ-CUTTING BY *X*-RAYS*

William Parrish† and Samuel G. Gordon‡

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Abstract

Methods for precise control of the cutting angles of quartz by *x*-ray measurements are described. The quartz must first be oriented by some other method (usually optically) before the *x*-ray method can be applied. The sense of direction of the cut is indicated by an arrow drawn on the outer surface of the test cut at the saw and this direction is preserved in making the *x*-ray measurement. The *x*-ray technique is an adaptation of the Bragg ionization chamber method and involves measuring the angle between the surface of the test cut or blank and an atomic reference plane parallel (or nearly parallel) to the surface. All measurements are direct, and require no computation. A Geiger-Muller tube operated in the proportional counter region is employed. Accuracy of the method is approximately ±1.5′, the measurement requires about 10–15 seconds, and is used by unskilled help. The procedures for calibrating the *x*-ray goniometer and measuring various types of cuts are described in detail. Methods involving precise angular adjustments of sections approxi-

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[*] Presented to American Society for *X*-Ray and Electron Diffraction, Ann Arbor, Michigan, June 10, 1943 under the title "*X*-ray techniques in the quartz oscillator industry."

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mately oriented by other methods and the use of reflection intensity differences of certain planes on either side of the optic axis for detecting usable portion of electrical twins and negative and positive directions from Z are described. The methods are applicable to other fields.

INTRODUCTION

The mass production manufacture of quartz oscillator-plates requires control of angles of cutting within quite narrow limits. The tolerances generally required for AT- and BT-cuts are ZZ′ ± 10′ and XX′ ± 15′ from the absolute values. The optical and etch techniques generally used in the preliminary orientation of quartz do not have this required precision. The *x*-ray techniques described below have proven to be the only economical and precise method known today for this purpose. This precision is required in modern oscillator-plates which are manufactured to meet high performance standards. Such properties as the temperature coefficient, frequency-thickness constant, coupling between various modes of vibration, etc., are critically dependent upon orientation and hence the general rule is not to depart greatly from the specified angles, in order to produce blanks with uniform properties.

Before the introduction of *x*-rays, manufacturers oriented by morphology or optical means and cut a test piece from the crystal. This test piece was fashioned into an oscillator and the frequency change with temperature determined. The correction to be applied to the saw was determined from these data but was generally inaccurate because of broadness of the turning point of the temperature-frequency curve, compound angular errors, and other factors. In any case, the test operation usually required about an hour during which time the saw remained idle. No real progress was made because there was no way to correlate precisely the physical porperties with orientation.

This state of affairs may be contrasted with that prevailing today when *x*-rays are used to make hundreds of thousands of measurements accurate to a few minutes every day by untrained help, taking 10 to 15 seconds for each measurement! In fact, this marks the first application of *x*-ray diffraction as an integral part of mass production manufacturing. The natural crystal faces, optical properties and light figures of etched sections serve for preliminary orientation to within a few degrees, but *x*-rays are used to assure precision in cutting to minutes. Indeed without *x*-rays, the mass production manufacture of quartz oscillator-plates with the present extraordinary standards of performance would be impossible.

The great usefulness of *x*-rays lies in the ability to use atomic planes within the quartz to bring a crystal to precise orientation when its natural faces have been sawn away. In actual practice a test sliver is cut on the saw and the angle between the surface and a reference atomic plane such as $(01\bar{1}1)$ for AT-cuts or $(20\bar{2}3)$ for BT-cuts is measured directly and the angular adjustments are then made at the saw. In all cases, however, preliminary orientation by some means must be provided to identify or set up the atomic planes close to the reflecting position.[1] The method also requires coordination between the saws and x-ray settings to preserve the sense of direction.

Acknowledgments. The Geiger-Muller counter tube used for x-ray measurements and its associated electronic circuit were developed by Dr. Herbert Friedman, Naval Research Laboratory, Anacostia Station, Washington, D. C. Without this timely development, the x-ray technique would not have become the invaluable tool for the manufacture of quartz oscillator-crystals. Dr. Friedman also prepared the x-ray powder spectrum of quartz shown in Fig. 11. The x-ray goniometer is based upon a design developed in collaboration with Bendix Radio Corp., Crystal Division, Baltimore, Md. The photographs were taken by Mr. John Derbyshire, N.A.P.

X-RAY APPARATUS

The x-ray apparatus in use by the quartz oscillator industry is an adaptation of the original Bragg ionization spectrometer. The great advantage of the instrument is its precision, as well as its simplicity of operation. Two commercial machines have been widely used (some 300 x-ray machines are in use in the crystal industry). The General Electric Company's apparatus employs a water cooled x-ray tube, drawing comparatively high current and uses an ionization chamber for detection. The North American Philips Co., Inc., manufactures a special unit, described below, (Figs. 1–4), for quartz crystal work in which a low current (3–4 ma.) air cooled x-ray tube is used with a high voltage, high sensitivity Geiger counter tube.[2] Both machines have two-window tubes and are equipped with an x-ray goniometer for each window.

Several types of x-ray crystal holders are available (Figs. 2, 5–8) for

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[1] Parrish, William and Gordon, Samuel G., Orientation techniques for the manufacture of quartz oscillator-crystals: *Am. Mineral.*, this issue; Cutting schemes for quartz crystals: *ibid.*

[2] The normal precautions in the use of x-ray apparatus should be followed as prescribed in National Bureau of Standards Handbook HB20, X-Ray protection, July 24, 1936, Supt. of Documents, Washington, D. C. The commercial apparatus is shock- and ray-proof. The on-off shutter must be closed before placing or removing the crystal from the holder. In some crystal plants, orientation of crystals mounted on jigs is attempted by x-rays. In several instances the operators get their fingers in the x-ray beam while making adjustments on the jigs. The carrying of dental films by the operator, while useful, does not indicate that the fingers are not getting in the beam.

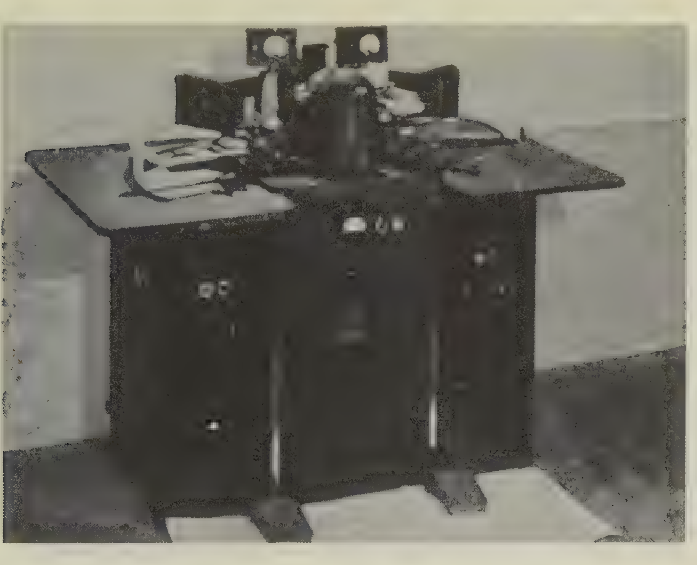


FIG. 1. North American Philips Co., Inc. *X*-ray machine for quartz crystals. Power supplies for each Geiger-Muller tube are in cabinets on either side of high voltage transformer and controls are contained in middle section.

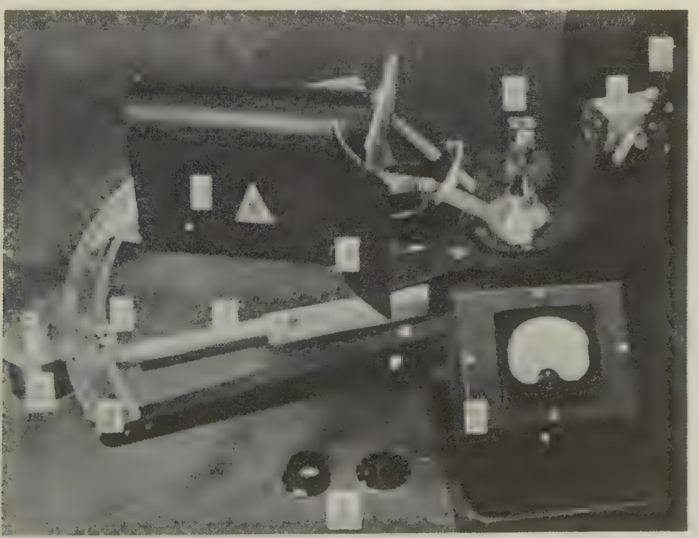


FIG. 2. *X*-ray goniometer. (A) Slit system, (B) on-off shutter, (C) crystal holder for measuring blanks, (D) collar for tightening crystal holder to goniometer arm, (E) 0–1 milliammeter for indicating *x*-ray reflections, (F) direct beam shield, (G) Geiger-Muller tube housing, (H) goniometer arm, (I) auxiliary scale, (J) scale graduated in 1° steps, (K) minute scale, (L) step and microswitch for changing voltage on G.-M.

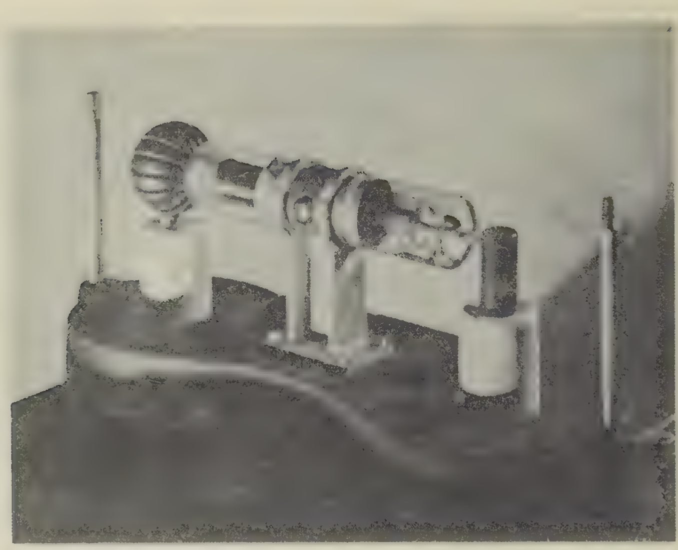


FIG. 3. Air-cooled x-ray tube with copper target and two Lindemann glass windows. Shadow shows position of protective housing.

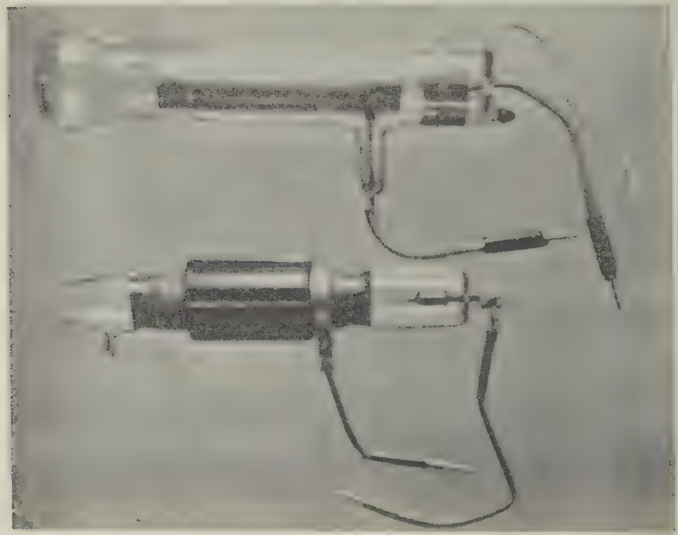
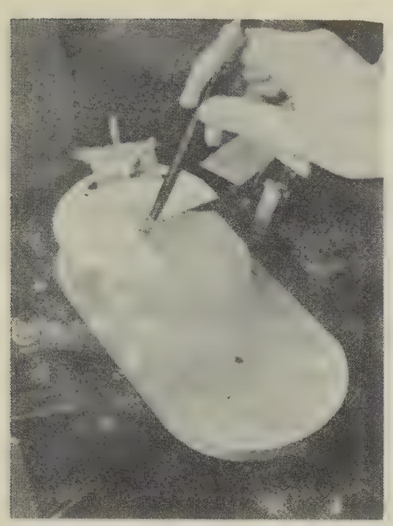


FIG. 4. Geiger-Muller tubes. Older type with thin bubble window above; newer type with Lindemann glass window and metal casing below.



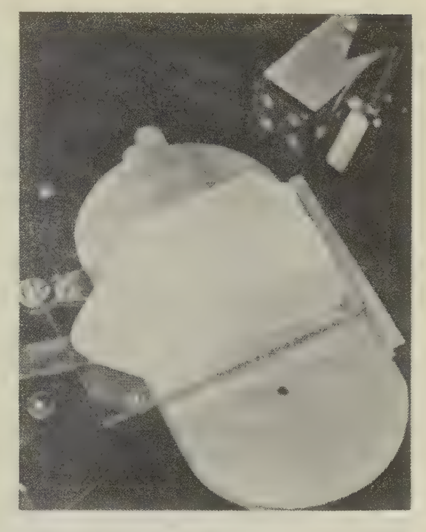
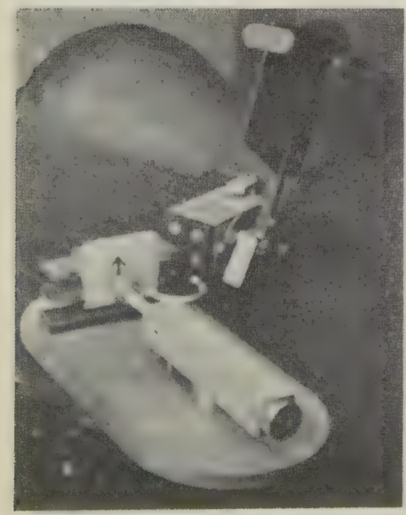


FIG. 5. *X*-ray table and marking guide for locating X- or Y-axes in Z-sections.
FIG. 6. *X*-ray table for measuring deviations of mounted crystal or X-block with respect to reference edge of glass mounting plate.



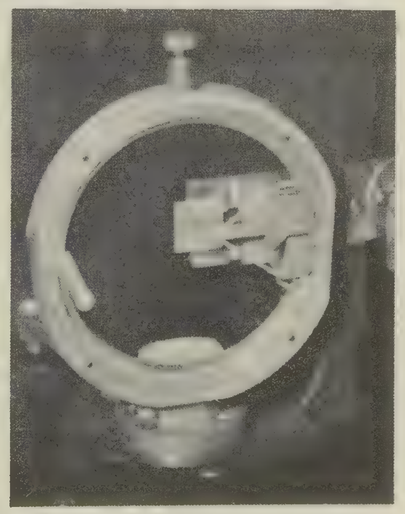


FIG. 7. *X*-ray table for measuring angular deviations of test cuts. Plunger arrangement allows rapid setting of quartz with irregular back.
FIG. 8. Rotating blank holder sometimes used for measuring ZZ′ and XX′ by rotating blank 90° after first measurement.

the various operations. In all, the test cut piece or blank is placed flush against the reference surface of the crystal holder of the x-ray goniometer. The crystal is held in place by a clip and spring. It is apparent that the anvil of the crystal holder must be perfectly flat to obtain accurate measurements. Although hardened tool steel, and sometimes boron carbide are used, the wear on the surface produced by constant use requires careful checking of the surface every few days. The crystal holder is locked to a ruled arm which is marked in degrees and a circular scale which reads directly to minutes. The Geiger tube setting is fixed and the holder with crystal rotated in the x-ray beam until maximum deflection of a milliammeter indicates the chosen atomic planes are in the position of maximum reflection. The setting is then read directly from the ruled arm and minute scale.

Geiger-Muller Counter Tube for X-Rays. It will be apparent from the description of the mass production techniques for manufacturing quartz crystals that film methods would have been totally inadequate. One can readily understand how production could not have reached its peak if the saw remained idle while a film of the test cut was being exposed, developed and measured. Fluorescent screen methods require a darkened room and have been used by few plants. Methods involving the use of pairs of reflection spots on fluorescent screens are confined only to the setting of quartz on a jig. Techniques for obtaining precise angular measurements by this means are not in use. Various ionization chamber techniques with sensitive galvanometers were tried but were generally not practical in the industry because of the small currents involved, vibration and humidity problems, critical adjustments, etc. The most successful device developed is the Geiger-Muller counter and associated electronic circuits developed at Naval Research Laboratory.[3]

The G.-M. tube, Fig. 4, is designed for high efficiency for the characteristic x-ray wave-length being used. It is filled with argon at nearly atmospheric pressure and a small amount of ethyl alcohol to make the tube self-quenching. This fast counter has extraordinarily high sensitivity—far greater than could be obtained with ionization chambers or film. The x-rays reflected from the crystal pass through a thin Lindemann glass window on the G.-M. tube and then through the gaseous area. About 85% of the Cu$K\alpha$ x-rays passing through the window are absorbed in the 10 cm. gas path. Each x-ray quantum produces a number of charged particles which cascade to form many others. These are collected at the anode of the G.-M. tube and recorded as one pulse. The chrome-iron cathode cylinder is sealed directly to the soft glass ends and the anode is a tungsten wire mounted in glass at the end of the tube. The wire is about 0.030″ diameter which permits it to remain suspended in the center of almost the entire length of the tube.

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[3] Friedman, Herbert; Kaiser, Herman F. and Christenson, Arthur L., Applications of Geiger-Muller counters to inspection with x-rays and gamma rays: *Jour. Am. Soc. Naval Eng.*, **54**, 177–209 (1942). For a general discussion of G.-M. tubes see Strong, John, *Procedures in Experimental Physics*, Prentice-Hall, Inc., New York (1938), Chapter VII, by H. V. Neher.

The sensitivity of the G.-M. tube is dependent upon the potential difference applied to the electrodes, since the tubes for quartz work are operated in the proportional counter region; i.e., just below the G.-M. tube plateau. Approximately 800 to 1200 volts is supplied by a constant voltage electronic D.C. power supply housed under the goniometer table;[4] a step switch with micro adjustments allows a rapid adjustment of the voltage. The exact voltage required will vary with the amount of gas in the tube, the reflecting power of the atomic plane, width of slits, slit to specimen to G.-M. distance, etc. In practice it is not necessary to adjust the voltage when making successive measurements on the same type of cut. The G.-M. tube is followed by a simple one-stage amplifier and a 3″ 0–1 milliammeter is used for detecting the current produced. The sensitivity of the apparatus is sufficient to detect all the commonly used atomic reference planes in quartz.

A series of experiments on BT-blanks showed that the x-ray measurements are reproducible to approximately $\pm 1.5'$. Greater accuracy may be obtained using the $(10\bar{1}1)$ atomic planes which give a sharper reflection than $(20\bar{2}3)$ and plotting a curve such as the one shown in Fig. 9.

Alignment Procedure. A piece of 0.0004″ thick nickel foil is attached to the window of the x-ray tube housing to absorb Cu$K\beta$. Two fixed slits each with an opening 1/32″ wide, 5/16″ high and 2″ apart and an on-off shutter are contained in a housing which is attached directly to the x-ray goniometer. The position of each slit may be adjusted by two screws each working against one of the edges of the piece of metal containing the slit. These are adjusted and aligned with the window on the x-ray tube housing by shifting the goniometer assembly. The G.-M. tube may be used in this alignment by removing the crystal holder from the path of the beam and placing a few pieces of plate glass in front of the window to reduce the intensity of the direct beam. The x-ray goniometer is then bolted to the x-ray machine table and the direct beam should then be at or very close to 0° on the scale. However, accuracy of this 0° setting is not important for the type of measurements generally used in quartz crystal work.

The crystal holder may be rotated independently of the goniometer arm by loosening the collar which locks it to the goniometer arm. The goniometer arm is set to a position on the scale which is convenient for reading (usually 0°). With the G.-M. housing set at the approximate 2θ for the desired atomic planes and the x-ray standard crystal in place, the holder is rotated by hand to a position of maximum reflection as indicated by maximum deflection on the milliammeter. The collar is then locked so that the goniometer arm can rotate the crystal holder and is accurately adjusted to maximum deflection. Then the G.-M. housing is moved until the milliammeter shows maximum deflection. The position of the G.-M. housing is not critical due to the wide scatter slit and the wide window on the G.-M. tube. The housing may be moved about $\frac{1}{4}°$ before the milliammeter will show a change from maximum deflection. The goniometer arm is then turned to maximum reflection and an auxiliary scale slipped on the goniometer scale and tightened in the new 0° position. The minute scale is set to 0′ by loosening the screw on top of the disc and rotating the scale by hand while holding the goniometer arm in place. The goniometer is now set up to give direct readings with respect to that of the standard crystal used in the alignment. If it is desired to make direct readings from a point on the scale other than the one given by the standard crystal, the goniometer arm is then rotated to the desired angle and the minute scale readjusted to 0′ for this new position. For example, if a $(10\bar{1}1)$ x-ray standard was used for the alignment and it was desired to measure directly the ZZ' angle of AT-cuts, the goniometer arm would be rotated $38°13' - 35°15' = 2°58'$ clockwise, the auxiliary scale moved to the new position and the minute scale reset to 0′.

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[4] Batteries have also been used. Since very little current is drawn, they practically have shelf life.

The crystal holder and G.-M. tube should be carefully checked for alignment in the horizontal plane with the slit by using a straight-edge and fluorescent screen. It is necessary that the opening in the crystal holder be wider than the incident x-ray beam to prevent scattering by the metal of the crystal holder which may cause a flicker of the milliammeter needle. The G.-M. tube should be set in the housing with the window close to the scatter slit and so positioned horizontally and vertically that the incoming x-ray beam passes along the tube without striking the electrodes.

X-RAY GONIOMETRY OF QUARTZ

The reflection of x-rays by a pile of parallel and equally spaced atomic planes is possible *only* when the Bragg condition is satisfied:

$$n\lambda = 2\ d \sin\theta, \text{ or}$$

$$\theta = \sin^{-1}\frac{n\lambda}{2d}.$$

When this condition is met, the angle of incidence, θ_i is equal to the angle of reflection θ_r. Therefore, to use a set of atomic planes for reference, their spacing must be known so that the collimated x-ray beam and the Geiger tube can be set at the angle 2θ apart, i.e., the sum of θ_i and θ_r, since the x-ray source is fixed and only the Geiger tube can be set. The spacing d of any set of atomic planes may be calculated from the precision measurements of the unit cell quoted by Wyckoff[5] using the formula

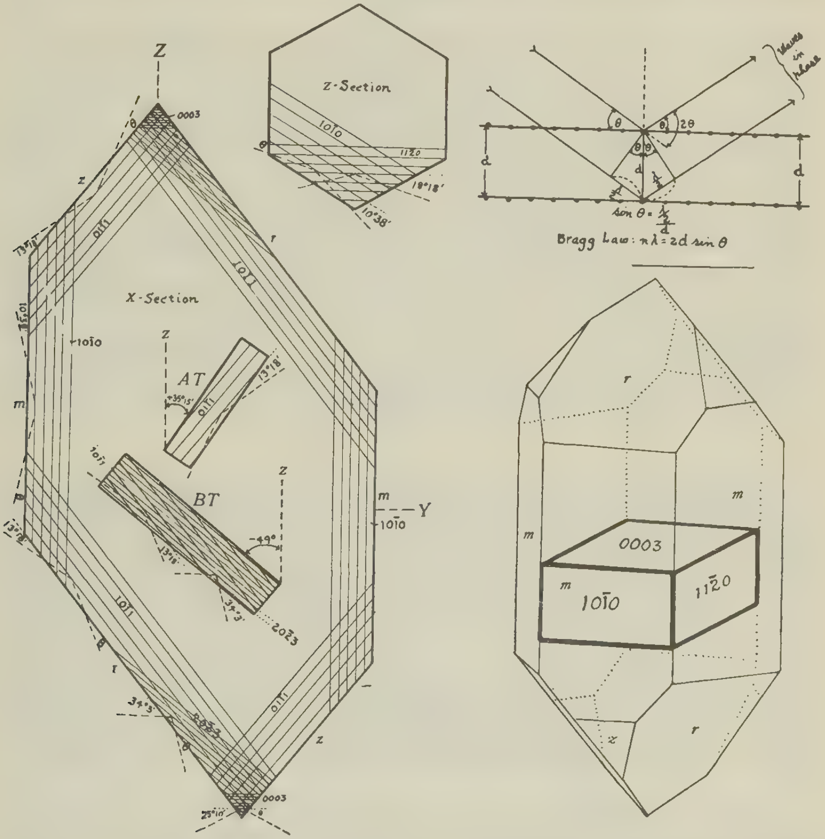

FIG. 9. Commonly used atomic planes for quartz measurement (left). Precisely cut x-ray standards (right) are useful in rapid calibration of x-ray goniometer.

$$d_{hkl} = \frac{a_o}{\sqrt{4/3\,(h^2+hk+k^2)+\dfrac{l^2}{(c_o/a_o)^2}}}$$

where $a_o = 4.9029$ Å, $c_o = 5.3933$ Å and $h\ k\ l$ refer to the indices of the plane. CuK x-radiation is filtered through 0.0004″ Ni foil to obtain monochromatic Cu$K\alpha$ radiation. The wave-length, λ, is

$$\frac{2\text{ Cu }K\alpha_1 + \text{Cu }K\alpha_2}{3} = 1.538674\text{ Å}.$$

X-ray data for some useful atomic planes of quartz are given in Table 1 and Fig. 9; more complete data are given by Bond and Armstrong.[6]

The x-ray reflections from various sets of atomic planes have different intensities and different natural breadths. For example, reflection from an AT-cut crystal using (01$\bar{1}$1) atomic planes is much stronger and sharper than a reflection from a BT-cut crystal using (20$\bar{2}$3) atomic planes (Fig. 11). The rhombohedral planes are the strongest reflectors in quartz, and in a recorded powder spectrum employing a Geiger tube (Fig. 10) they are 13 on an arbitrary intensity scale in which (20$\bar{2}$3) is 2. The width of the reflection at half the height of finely lapped and etched single quartz crystals cut parallel to the atomic planes, as measured on the Philips crystal analysis unit, is approximately 20′ to 25′ for (10$\bar{1}$1) and 40′ for (02$\bar{2}$3).

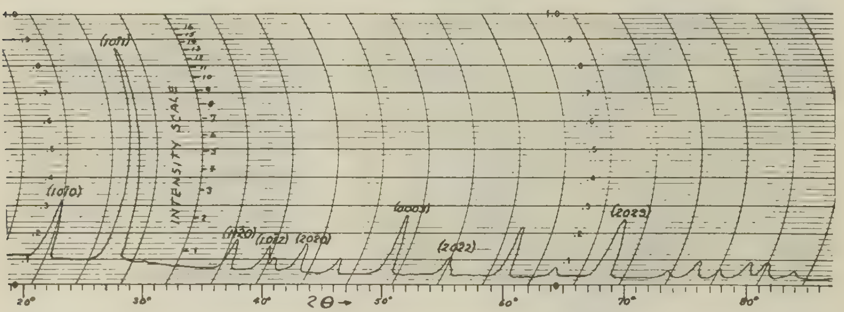

FIG. 10. X-ray powder spectrum of quartz automatically recorded on new Geiger-Muller tube spectrometer. Arbitrary intensity scale. (Prepared by Dr. Herbert Friedman, Naval Research Laboratory.)

Choice of Atomic Reference Planes. It is advisable to choose an atomic plane for reference which is close to parallelism with the surface of the cut. In this way, absorption is reduced to a minimum, reflections are obtained even if the test cut is quite far off angle and errors due to im-

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[5] Wyckoff, Ralph W. G., *The Structure of Crystals*, supplement to 2nd ed., p. 26, Reinhold Publishing Corp., New York (**1935**).

[6] Bond, W. L. and Armstrong, E. J., Use of x-rays for determining the orientation of quartz crystals: *Bell Syst. Tech. Jour.*, **22**, 293–337 (1943).

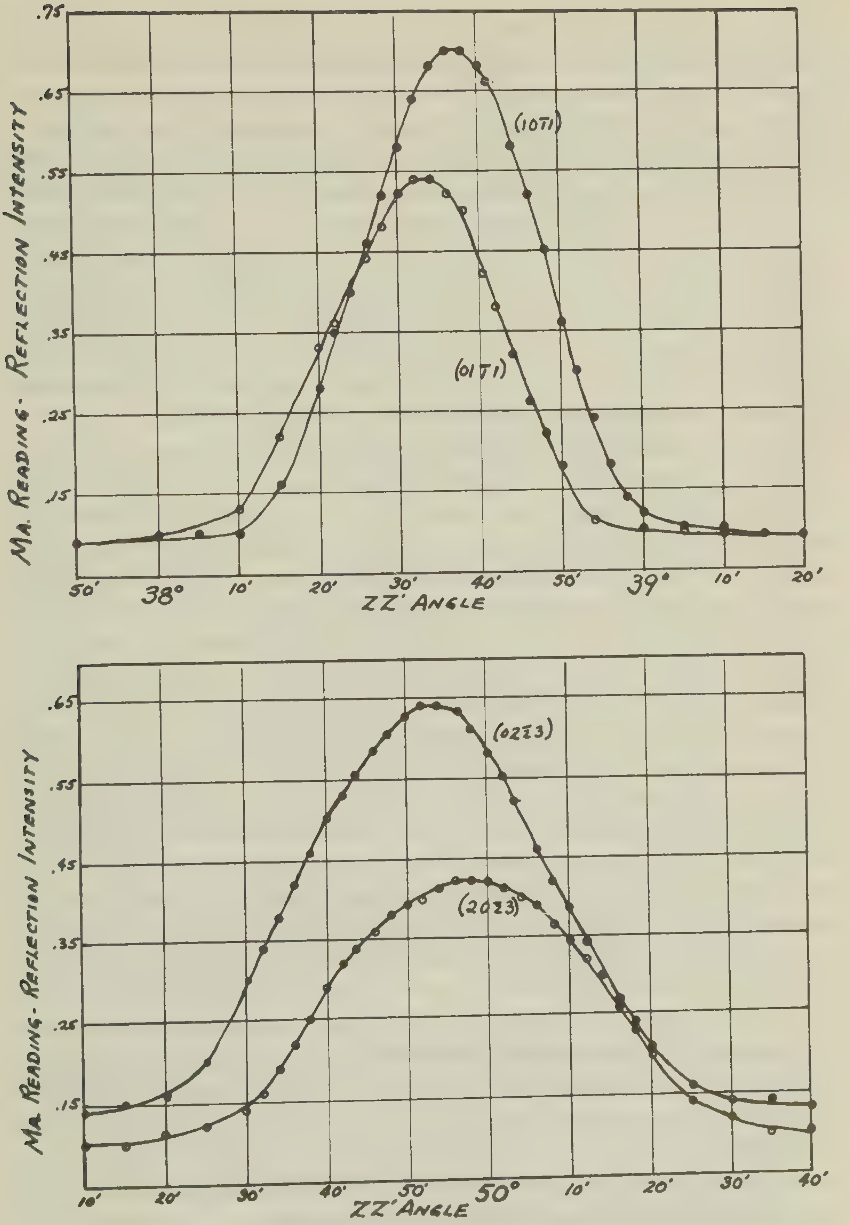

FIG. 11. *X*-ray reflections from electrically twinned etched wafers. Stronger reflection is obtained from $(10\bar{1}1)$, the useless side for AT-wafers and from $(02\bar{2}3)$ the useless side for BT-wafers. $(10\bar{1}1)$ and $(01\bar{1}1)$ give much stronger and sharper reflections than $(20\bar{2}3)$ and $(02\bar{2}3)$. Voltage on G.-M. tube increased for latter to obtain approximately same ma. deflection. Both wafers set in XX′ position. Twins also show deviation of approximately 5′.

proper marking of the X-axis direction are reduced to a minimum. The latter is particularly important in precise measurements of AT- and BT-crystals blanks. For example, if $(10\bar{1}1)$ atomic planes are used to measure the ZZ′ angle of a BT-crystal, the angle between the physical surface of the blank and $(10\bar{1}1)$ will be 49°20′ (nominal) $-$ 38°13′ $=$ 11°07′. This is the maximum error possible in measuring ZZ′ when the X-axis is 90° from its proper position. Intermediate-errors are obtained for intermediate settings of X. The true ZZ′ angle is measured only when X is vertical and rotation of the blank is about this axis. If $(20\bar{2}3)$ is used for the reference atomic plane for BT-cuts, the error introduced by an incorrect setting is very small; 24′ would be the maximum for a complete 90° error in setting X; in practice, the error in this case is negligible. The commonly used reference planes for the various cuts are listed in Table 1.

X-Ray Standards. Thick crystals (about 0.100″) with marked reference edges cut parallel to the reference atomic planes are useful in setting up and calibrating the *x*-ray goniometer. The crystal is machine lapped and etched to assure freedom from twinning and to sharpen the *x*-ray reflection.[7] The angular-view stauroscope conventions are described in the previous paper by the writers. A line is ruled on the standard to indicate X and an arrowhead to show Z. The following planes are useful for standards: (0003), $(10\bar{1}0)$, $(11\bar{2}0)$, $(10\bar{1}1)$, and $(02\bar{2}3)$. Note that $(10\bar{1}1)$ instead of $(01\bar{1}1)$ and $(02\bar{2}3)$ instead of $(20\bar{2}3)$ are used for standards *only*, to take advantage of the stronger reflections of these planes. It will be apparent that if the standard has been cut exactly parallel to the atomic plane, the *x*-ray reading will be the same for any position of the standard. If the standard is not exactly parallel to the atomic plane, and the X or Z directions are known either from the remains of a natural face or by careful stauroscoping, the exact position on the goniometer scale for that atomic plane may still be determined. One *x*-ray reading is obtained with the arrow up (for rotation about a crystallographic axis), and another from the same surface with the arrow down (180° apart). The atomic plane is half-way between these two readings. For direct settings for AT- and BT-cuts, accurately cut standards at the desired angle to the atomic plane may be cut and X and Z must be marked to determine the correct direction. A standard parallel to the desired atomic plane may also be used by setting the goniometer scale to zero for this reading by rotating the minute arm to the desired ZZ′ angle, and resetting it and the auxiliary scale to this new zero point.

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[7] Manning, K. V., The effect of etching on the rocking curve width of calcite crystals: *Phys. Rev.*, **43**, 1050 (1933).

TABLE 1. CRYSTALLOGRAPHIC AND *X*-RAY DATA FOR QUARTZ

Atomic Plane (*hkīl*)	Crystal Face (Dana)	Crystallographic Angles ϕ	ρ	ZZ′	Used for Measuring Plane	Angle Between Plane of Cut and Atomic Plane	G.M. Tube Setting 2θ*	Relative Intensity*
0003	*c*	00°00′	00°00′	90°00′	Z	0°00′	50°40′	.5[a]
$10\bar{1}3$		“	22°57′	−67°03′			55°22′	45.
$01\bar{1}3$	*ω*	“	“	+67°03′			“	2.7
$10\bar{1}2$	*d*	“	32°25′	−57°35′	FT	− 0°35′	39°30′	28.
$01\bar{1}2$	*π*	“	“	+57°35′			“	85.
$20\bar{2}3$		“	40°16′	−49°44′	BT[b]	+ 0°24′	68°10′	32.
					DT	− 2°14′		
$02\bar{2}3$		“	“	+49°44′	GT[c]	+ 1°46′	“	63.
$10\bar{1}1$	*r*	“	51°47′	−38°13′	BT[b]	−11°07′	26°40′	100.
$01\bar{1}1$	*z*	“	“	+38°13′	AT	+ 2°58′	“	75.
					CT	+ 0°13′		
$20\bar{2}2$	*r*	“	“	−38°13′			54°54′	26.
$02\bar{2}2$	*z*	“	“	+38°13′			“	26.
$30\bar{3}2$	*j*	“	62°18′	−27°42′			75°42′	19.
$03\bar{3}2$		“	“	+27°42′			“	27.
$50\bar{5}2$		“	72°31′	−17°29′			143°24′	.25
$05\bar{5}2$		“	“	+17°29′			“	23.
$30\bar{3}1$	*M*	“	75°18′	−14°42′			68°20′	40.
$03\bar{3}1$	*M′*	“	“	+14°42′			“	1.4
$10\bar{1}0$	*m*	“	90°00′	00°00′	Y	0°00′	20°52′	24.
$20\bar{2}0$	*m*	“	“	“	Y	“	42°28′	28.
$11\bar{2}0$	*a*	30°00′	“	“	X	“	36°34′	13.[d]

* Data in these two columns from Bond and Armstrong, *Bell Syst. Tech. Jour.*, **22,** p. 303, (1943); these are *estimated* values and do not check well with Figure 11.

[a] This value is in error; (0003) has about the same reflecting power as ($20\bar{2}3$).

[b] Based on ZZ′ = −49°20′.

[c] Based on ZZ′ = +51°30′.

[d] Estimated from Figure 11 by comparison with ($10\bar{1}0$).

The values in brackets (}) in the last column represent *x*-ray reflection differences useful for determining (+) or (−) angle from Z.

X-RAY MEASUREMENTS OF CUTTING ANGLES

The use of the *x*-ray technique makes it possible to rapidly and accurately determine the corrections that must be applied to the saw table to bring the cut within the desired tolerance. The following is a description of the most commonly used measurements employed in the manufacture of quartz oscillator-crystals. The marking conventions for correlating the x-ray goniometer readings with saw table corrections are summarized in Table 2.

TABLE 2. MARKING CONVENTIONS FOR CORRELATING X-RAY GONIOMETER AND SAW TABLE

	Horizontal Angle at Saw Table (ZZ′)		Vertical Angle at Saw Table (XX′), AT- or BT-Type Cut	
	AT-Type Cut	BT-Type Cut	Facing Saw, Cut from left side of crystal	Facing Saw, Cut from right side of crystal
Test Cut Wafer	↑	↑	←	→
X-ray Standard or Blank	◁	▷	△ or ▽	△ or ▽

X-Ray Measurement of X-Plane. The desired cut is one parallel to the $(11\bar{2}0)$ atomic planes but the following discussion may be applied to any cut parallel to any atomic plane by simply setting the Geiger tube for that particular reflection.

A vertical arrow pointing upward is marked on the outer surface (side away from the blade) of the test piece to preserve the sense of direction of the cut.[8] Burrs must be removed from the fresh cut surface of the test piece to prevent errors in x-ray measurement. The following discussion refers to both right and left goniometers. By using the terms *clockwise* and *counterclockwise* there is no ambiguity as to direction; it will be noted that clockwise on the right goniometer brings the goniometer arm towards the observer at the front of the machine, and away from him on the left goniometer. The goniometer is set with a $(11\bar{2}0)$ standard so that the scale reads 0°0′ when $(11\bar{2}0)$ atomic planes, $2\theta = 36°34'$ for Cu$K\alpha$, are in a position of maximum reflection.

The test piece is placed flush against the reference face of the x-ray crystal holder with the fresh cut side toward the x-ray beam and the opposite side with the arrow pointing upward is facing the operator. The goniometer arm is rotated clockwise or counterclockewise until the needle of the Geiger tube milliammeter shows a peak reading. If the cut were truly parallel to $(11\bar{2}0)$ the goniometer reading would be 0°0′. In general there will be a deviation, either clockwise or counterclockwise. To show the nature of the correction, let us assume that the peak reading was at 1°35′ clockwise from 0° on the scale. At 0° the test cut was *not* parallel to $(11\bar{2}0)$ and it was necessary to correct the setting by 1°35′ clockwise to bring it parallel to the $(11\bar{2}0)$ planes. It is apparent that the quartz on the saw table corresponds to the first position at the x-ray goniometer, 0°, and that it is therefore necessary to make exactly the same correction, in the same direction, 1°35′ clockwise, at the saw. It is also apparent that there has been no change in position of the quartz or the test cuts between the saw and the x-ray apparatus as is evinced by the procedure of marking the vertical arrow on the test cut, and preserving this direction on the x-ray crystal holder.

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[8] In some of the earlier x-ray measurements of quartz, while it was appreciated that this sense of direction must be preserved, instead of marking the test cut at the saw, it was stauroscoped. This introduced errors in making the corrections because the stauroscope markings were not necessarily coincident with the axes of rotation of the saw table.

To test the vertical angle of the saw table, the test piece is rotated 90° so that the arrow is now horizontal and another position of maximum reflection obtained. If the test cut has been made from the right side of the crystal (when facing the saw) the arrow points to the right; if the test cut was made from the left side of the crystal, the arrow should be pointed to the left to obtain a direct reading of the correction that must be applied to the vertical plane of the saw table. An alternative procedure is to point the arrow in the same direction for either right- or left-side test cuts and correct the former in the same direction and the latter in the opposite direction to that indicated by the x-ray reading.

X-Ray Measurement of AT-Test Cuts. AT-wafers are cut at an angle of +35°15′ from the optic axis, or at an angle of −2°58′ from the z $(01\bar{1}1)$ plane which is at +38°13′ from Z. It is therefore convenient to check the ZZ′ angle (horizontal angle of the saw table) of cut with $(01\bar{1}1)$; i.e., the plane of the sawed surface should be −2°58′ from the $(01\bar{1}1)$ atomic reference planes within the quartz wafer. It will be obvious that x-rays cannot distinguish between a plane cut at −2°58′ (ZZ′ = 35°15′) from one cut at +2°58′ (ZZ′ = 41°11′) to z $(01\bar{1}1)$ so that it is essential to transfer the sense of the cut from the saw to the x-ray machine.[9]

The most convenient procedure is to preset the goniometer by means of either a precisely cut AT or $(10\bar{1}1)$ atomic plane standard as described in the section on x-ray standards. If an AT-standard is used it should be positioned with the line indicating the electric axis vertical and the arrowhead indicating the optic axis direction to the left as determined with the angular view stauroscope. (The stauroscope markings on the standard are not to be confused with the markings of the test cut.) Test wafers, like all other test cuts, are marked at the saw table with a vertical arrow pointed upward on the side of the test wafer opposite the fresh cut. The wafer is set in the crystal holder so that the *x*-ray beam strikes the fresh cut surface with the arrow in view of the operator. The arrow is pointed upward to measure the correction required for the horizontal plane (ZZ′) of the saw table. If the goniometer reading is not 0° (corresponding with the *x*-ray standard setting) an angular correction of the same amount and in the same direction, clockwise or counterclockwise, is marked on the wafer to be duplicated at the saw.

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[9] One way of distinguishing them is to set the goniometer arm to zero for $(20\bar{2}3)$, ZZ′ = 49°44′. If the cut is at +2°58′ from $(01\bar{1}1)$, a reflection will be obtained at 8°33′ or at 12°59′ if cut at −2°58′ when the X-axis is vertical. For reasons mentioned above, an atomic reference plane closer to the surface is required for precision measurements.

Since the surface of the wafer is inclined to the atomic reference planes, the horizontal and vertical angle corrections are read from different points of the *x*-ray goniometer scale. Thus XX′ 38°13′ − ZZ′ 35°15′ = 2°58′ apart for AT-cuts.[10] To measure the vertical angle, XX′, the goniometer is set by means of an *x*-ray standard with its zero point corresponding to 38°13′ for maximum reflection from $(01\bar{1}1)$. In this setting the arrow is horizontal and if XX′ = 0°, the atomic planes are parallel to the edge of the wafer. The arrow is pointed to the right if the test cut was made from the right side of the crystal, and to the left, if cut from the left side, to obtain a direct reading of the vertical angle correction and direction that must be duplicated at the saw table.

X-Ray Measurement of BT-Test Cuts. The turning point of the temperature-frequency curve varies with ZZ′ angle and in BT crystals, angles varying from −49°00′ to −49°30′ are used, depending upon the desired properties. For this discussion we will assume a selected angle of −49° 20′. BT-wafers are cut at an angle of +11°07′ from the *r* $(10\bar{1}1)$ major rhomb face which is at −38°13′ from Z. As described above, it is desirable to use an atomic reference plane close to parallelism with the cutting surface. The best planes to use are $(20\bar{2}3)$ which are but +24′ from the plane of the cut $(Z\wedge(20\bar{2}3) = 49°44′)$.

Horizontal and vertical angular measurements and corrections are made in the same way as described above for AT-wafers, with these two exceptions: (1) the BT or $(02\bar{2}3)$ standard is placed in the *x*-ray crystal holder with X-axis vertical and optic axis direction to the *right;* (2) the vertical angle reading, XX′, is made 24′ from the ZZ′ reading.

X-Ray Measurement of AT- and BT-Wafers and Blanks. It is often necessary to check the angles of a stock of blanks or wafers to determine ZZ′ and XX′. *X*-ray measurements of all blanks prior to lapping is part of the inspection scheme to eliminate those not meeting the rigid angular specifications. Although *x*-ray measurements are used to control the cutting angles, errors creep in due to drift of the blade, inaccurate ways of the saw, etc. The *x*-ray measurements are preceded by orientation of the blank or wafer in the angular-view stauroscope, by means of which the optic axis direction, indicated by an arrowhead, and the direction of the electric axis, indicated by a line, are identified. This stauroscope pre-orientation permits setting of the blank in the *x*-ray beam in a position comparable to its orientation in the quartz when it was cut and avoids all ambiguity as to whether XX′ or ZZ′ is being measured or if the reading is "high" or "low." Blanks cut from wafers with reference edges (X-planes in the X-block method) and laid out by means of a rubber stamp with marked stauroscope directions need no further stauroscoping. Furthermore, if they have been properly squared the edges of the blank are parallel to X and Z′.

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[10] Where large production is required it is advisable to set up one goniometer to measure ZZ′ directly and the other XX′.

AT-cuts may be checked by reference to the $(01\bar{1}1)$ atomic planes with the Geiger tube set at 26°40′. The ZZ′ angle is measured by placing the blank in the holder with the arrow-head (Z direction) pointing to the *left* (marked side of blank toward operator and away from *x*-ray beam); rotation is thus about the X-axis. The XX′ angle is measured by turning the blank 90° with the arrowhead pointed either up or down.

BT-cuts are checked by reference to the $(20\bar{2}3)$ atomic planes with the Geiger tube set at 68°10′. The ZZ′ angle is measured by placing the blank in the holder with the arrow-head (Z direction) pointing to the *right*. XX′ is measured with the arrowhead pointed either up or down.

The *x*-ray goniometer is accurately preset to 0°0′ with an *x*-ray standard and must be reset or read from different points of the scale when switching from ZZ′ to XX′ measurements as described above.

PRECISE ANGULAR ADJUSTMENTS WITH *X*-RAYS

In addition to being an invaluable technique for measuring angles of cuts, *x*-rays are used to adjust cut wafers on jigs for angle correction by lapping when required; for determining the error in crystals set up with the mounting stauroscope prior to cutting so that the saw can be adjusted before making a test cut; and in the precise location of X- and Y-axes in Z-sections approximately oriented by other methods.

X-Ray Adjustment of Wafer Angle Correction Jigs. This procedure was introduced to correct wafers cut by the flat-lay method. Its purpose is to bring a wafer to adjustment so that its surface can be ground to the proper plane. The wafers are individually cemented to a tilting plate on one end of a rather expensive, specially designed, cylindrical jig, with a chamfered edge of the wafer parallel to two of the four adjusting screws at the other end of the cylinder. Each pair of screws tilts the wafer at right angles to the other pair. The jig is set in a special preset chuck in the *x*-ray beam and the wafer adjusted until *x*-ray reflection shows that the plane of cut desired is exactly perpendicular to the axis of the cylinder.

The jig is then rotated 90° and the adjustment repeated. A number of jigs are locked to the rotating arm of an ultralap which quickly trues the surface. The wafers are then recemented with the trued face down and the other side corrected with the ultralap.

X-Ray Presetting of Mounted Quartz Crystals. In large scale production, where every extra cut soon involves enormous additional costs, every attempt must be made to reduce the number of test cuts. This can be effectively done by x-ray measurement of the accuracy of the mounting and subsequent correction of the saw table before the first test cut is made. This does not eliminate the necessity of making test cuts, because in actual practice it is extremely difficult to preset within the precision usually required, but there is better chance for the accurate adjustment of the crystal for cutting with only one test cut.

The quartz crystals are mounted on a prism or rhombohedral (major or minor) face with the optic axis parallel to the long reference edge of a rectangular glass plate, utilizing the mounting stauroscope. The mount is set in the x-ray beam with the short reference edge of the glass in contact with the reference bar of the x-ray platform (Fig. 6). With the Geiger tube set at 36°34′ for reflection from $(11\bar{2}0)$ planes, the goniometer arm is rotated to a position of maximum reflection. If the goniometer scale has been preset to 0° by means of a precisely cut standard, the deviation of the optic axis of the quartz with respect to the short reference edge of the glass plate can be read directly. The angular correction, and its clockwise or counterclockwise direction, are marked on the glass plate so that the setting may be duplicated at the saw.

The same procedure may be followed in presetting X-blocks prior to wafering, by using the $(10\bar{1}0)$ atomic planes and in setting up Z-sections prior to cutting X-blocks or Z-bars.

Precise Location of X- and Y-Axes in Z-Sections. In cutting large quartz crystals, the strategy calls for the preliminary sawing into Z-sections, and then into X-Sections (or if the quartz has large flawless areas, into Y-bars). The general direction of the X- or Y-axes can be determined from the light figures of the etched Z-sections, by optical twinning if present, or by the other methods described. An accurate location may then be obtained by x-rays. The Z-section is placed on a platform (Fig. 5) on the x-ray goniometer with the approximate direction determined from the light figure in the approximate reflection position. The goniometer arm (and platform) is rotated until a peak milliammeter reading indicates the desired atomic planes are in the position of maximum reflection. This direction is ruled on the section by means of a right angle marking guide previously set with a standard to determine X with $(10\bar{1}0)$ or Y with

$(11\bar{2}0)$. Since these two axes are perpendicular to each other, the right angle permits marking one axis if the other cannot be found due to irregularity of the edge of the Z-section.

X-RAY APPLICATIONS USING REFLECTION INTENSITY DIFFERENCES

The marked differences of reflecting power of certain atomic planes on either side of the optic axis has made it possible to apply *x*-rays to the determination of usable portions of electrically twinned wafers, and the negative or positive cutting directions from Z. These methods are not widely used because simpler tests more adaptable to production routine are available.

X-Ray Determination of Usable Parts of Electrically Twinned Wafers. A test for the usable portion of electrically twinned wafers can be readily made by *x*-rays.[11] It is based on the difference in intensity of reflection of *x*-rays from r $(10\bar{1}1)$ and z $(01\bar{1}1)$ planes. The etched AT-wafer is set in the *x*-ray beam and the goniometer adjusted to a peak reading. The micro-adjustment of the voltage on the Geiger tube is set so the reading is at approximately mid-scale on the milliammeter. The shutter is closed and the wafer pushed horizontally to bring an area on the opposite side of the twin boundary into the *x*-ray beam. The intensity of reflection will change and be higher if on the side of the twin boundary reflecting from r $(10\bar{1}1)$ as shown in Fig. 10. The test is also applicable to etched BT-wafers with the Geiger tube set at 68°10′ for reflection from $(20\bar{2}3)$. In this case the lower reflection is from the side nearly parallel to the major rhomb. Thus in AT- and BT-wafers, that portion of the electrical twin with the weaker *x*-ray reflection is the usable side. The position of the two individuals is also shifted 2′ to 5′.

X-Ray Method of Determining Negative and Positive Directions from Z. This method was an early one for this purpose, and depends upon the fact that the $(30\bar{3}1)$ reflects strongly but $(0\bar{3}31)$ does not reflect at all.[12] The Geiger tube is set at 68°20′ and a wedge of 14°42′ is used to tilt an untwinned etched portion of the Z-section so that $(30\bar{3}1)$ is vertical. If no reflection is obtained, the Z-section is turned upside down on the wedge platform.

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[11] This method was developed by Dr. Dominick D'Eustachio, "A method for determining which portion of a twinned wafer should be used": *Tech. News Bull. No. 6*, Signal Corps Ground Signal Agency (1943).

[12] This reflection asymmetry was pointed out by R. E. Gibbs, Structure of quartz: *Proc. Roy. Soc. (London)*, **110,** 443–455 (1926). See also Broughton, William W., Piezoelectric apparatus: *U. S. Patent Off.* No. 2,151,736, Mar. 28, 1939 and Bond, W. L. and Armstrong, E. J., *op. cit.*

POSSIBLE APPLICATIONS IN OTHER FIELDS

The x-ray techniques described here may be developed for application in other fields. It has been used in the cutting of calcite and small diamond crystals for wire dies have been oriented with the technique. The orientation of sapphire for jewel bearings and other applications seem apparent.

The use of the G.-M. tube for diffraction work is a field which deserves the attention of x-ray crystallographers.[13]

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[13] Friedman, H., Geiger-counter spectrometer for industrial research: *Electronics*, **18,** 132–137 (1945).

CUTTING SCHEMES FOR QUARTZ CRYSTALS

Samuel G. Gordon* and William Parrish†

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Abstract

The various schemes for cutting small, large and defaced quartz are critically reviewed. In the single mount or direct wafering methods, the quartz is set up once-for-all and wafered. These methods are favored by "production" men because they involve a minimum number of operations. They usually require orientation by hand and polarity determinations and the use of transfer jigs. The cutting accuracy and yield are low.

In the X-block method, the crystal is mounted on a prism or rhomb face and a pair of X-planes cut. After etching, the block is oriented by parallelogram light figures, which are fool-proof, and wafered. The method has greater precision and permits the maximum utilization of electrically and optically twinned quartz. It does not require the use of jigs, the cutting accuracy is high, and it allows an easily visualized set of orientation rules and accurate control of the dicing angle. Flawed areas may be painted out to avoid useless sawing and processing. A pair of Z-planes is first cut on defaced quartz, Y determined with the triangular light figures and then handled as faced quartz.

Large faced or defaced quartz (greater than 1000 gms.) is cut into thick Z-sections and then into X-blocks which are wafered. If the quartz is of exceptional quality, it is cut into thin Z-sections, the width of the desired blanks, then into Y-bars which are cut into blanks.

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INTRODUCTION

A surprising variety of orientation techniques and cutting schemes have been devised for cutting quartz crystals. In this paper, only the strategy of the cutting schemes and the more successful procedures will be described.

Since the majority of the crystal cuts are in the prism-rhombohedral zone and at angles near either the *r* or *z* rhombohedral faces, it will be seen that there are two general geometrical approaches to arrive at the cutting plane.

(1) By mounting the crystal on a *prism edge* and rotating it about the now vertical X-axis to the desired cutting angle.

(2) By mounting the crystal on a *prism face* and tilting it (rotating about a horizontal X-axis) until the cutting plane is parallel to the saw blade.

To some extent, the procedures are determined by the available sawing equipment. There are two general types of saws:

(1) Converted milling machines which have little or no adjustment and require that the quartz be *preset* before it is placed on the saw; and

(2) The Felker type[1] which have graduated tables with horizontal and vertical angle movements and permit angular adjustments of the crystal as indicated by x-ray measurements of a test cut piece.

With respect to cutting quartz, there are two opposed schools of thought:

(1) The single mount or direct wafering set up, in which the quartz is preset in a cutting position once and for all, and sliced into wafers. These include flat lay cutting, "baloney slicing," vertical glass mount, transfer jig method and the dop method.

(2) The preliminary cutting of a precise X-plane, upon which the crystal is remounted and then sliced into wafers as in the X-block, wafering method.

The direct wafering approach is favored by most "production" men because it involves a minimum number of operations. If quartz were an untwinned and flawless substance, and were it really possible to orient it with precision once and for all, such a short cut would be justified.[2] In actual practice a large number of saws are required, and the efficiency is low for once a crystal is set up, good and bad portions are sliced, since it is not possible to discriminate between twinned and untwinned, or flawed and usable portions at the saw. Furthermore, the cutting accuracy is low due to the instability of the jigs and irregularity of the entering surface.

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[1] Including converted drill presses to which rotating tables, graduated circles and tilt table platforms have been added.

[2] Tilts of the saw table are often made in an effort to make angular corrections in three directions. Users of a universal stage will appreciate the problem involved and the results likely to be obtained by a saw operator who could hardly be expected to know that a crystallographic direction of the quartz must be brought parallel to the axis of rotation before a tilt is attempted. Elaborate arc movements within yokes carrying the crystal, gimbals and the like have been devised to correct the "once-for-all" set-ups.

The second approach, the X-block method, involves an additional cutting, etching, reorientation and cementing operation.[3] This is justified by (1) greater precision, since a true X-plane is cut at the start to assure that the X-axis is in the plane of the blanks; (2) foolproof orientation by means of the parallelogram light figure on the etched X-block, thus eliminating hand and polarity determinations; (3) a maximum utilization of electrically and optically twinned quartz; (4) elimination of sawing of useless flawed and twinned parts of a crystal; (5) generally only the horizontal angle need be adjusted at the saw—the vertical angle correction,

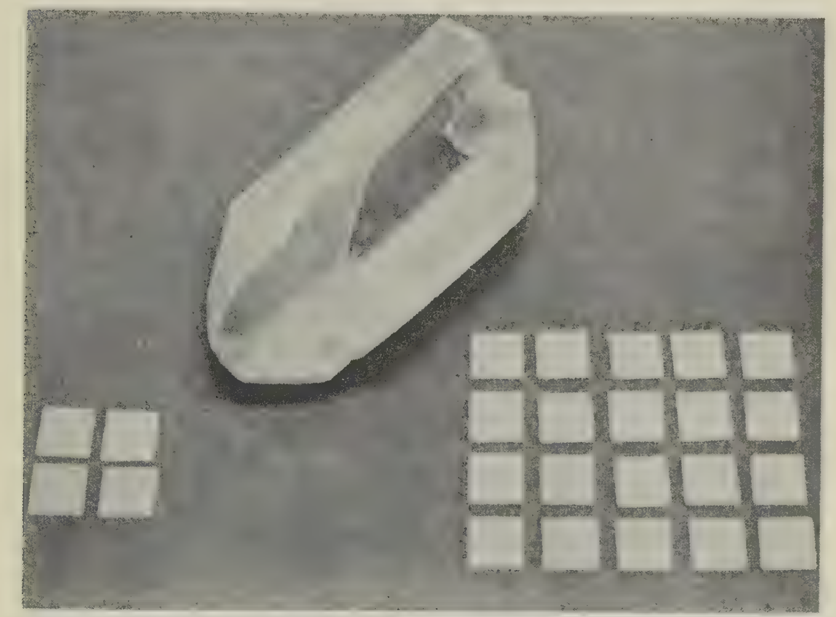

FIG. 1. The yield in most of the crystal plants in the country in 1943 of BT crystals varied from 20 to 4 units shipped per pound of raw quartz cut. On a weight basis this yield is considerably less than 1%.

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[3] In spite of these additional steps, manufacturers using this method use fewer saws and employees and produce more crystals per pound of quartz per man per day than those using the direct wafering methods.

if required, is small; (6) the cutting accuracy in wafering is greater because the blade enters perpendicular to a smooth surface and there are no jigs to reduce stability; (7) an easily visualized set of rules for orientation and correlation of *x*-ray goniometer readings with saw table rotations; (8) accuracy in control of the dicing angle with respect to the reference X-planes on the wafers. The method is simple, straightforward and does not require the use of jigs. After an additional preliminary operation defaced quartz may be processed in exactly the same way.

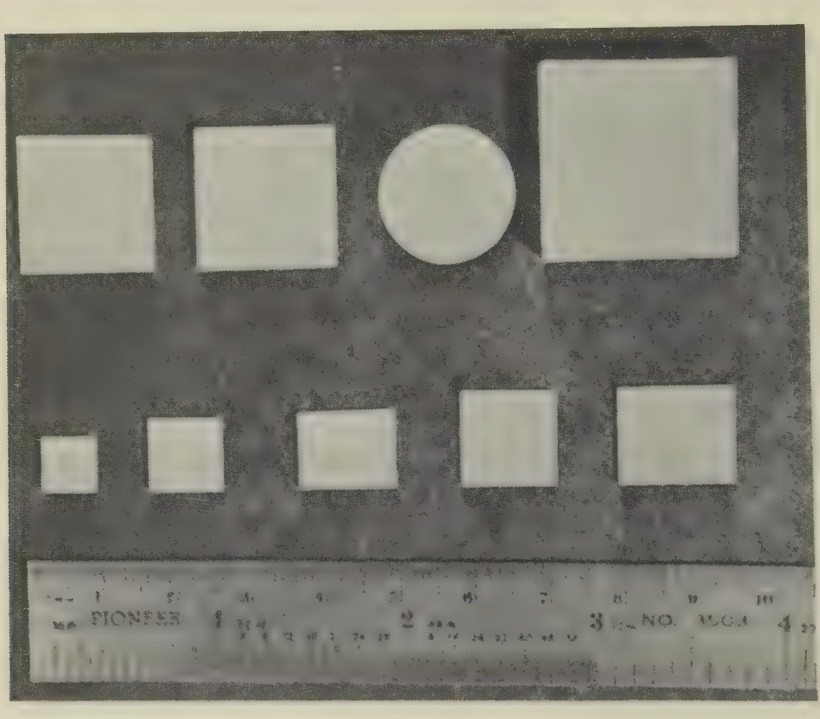

FIG. 2. Finished oscillator-crystals vary in size from approx. ¼″ to ¾″ square. Tendency is towards smaller sizes. Circular plates are used mainly abroad. Large plate in upper right is for ultrasonic work.

Although achievements were considerable in meeting military requirements for millions of crystal units a year, the efficiency of the industry calculated on the basis of the number of finished accepted crystals per pound of raw quartz cut was very low, but is now gradually increasing. The yield in 1943 for BT crystals for all qualities of raw quartz used in most of the crystal plants in the country varied from approximately 20 units to 4 units or even less (Figs. 1 and 2) shipped per pound of quartz cut. From the point of view of the weight lost, this represents an efficiency of *considerably less than* 1%. The largest loss is due to the thickness (0.045″ to 0.070″) of the diamond blades which cause losses up to 70%–80% in the wafering operation.

Acknowledgments. Many of the procedures here described are based upon methods developed in the quartz crystal industry. Mr. J. N. Bagwell, Commercial Crystal Co., Lancaster, Pa., collaborated in the early development work on the X-block method, and Miss Judith Weiss, Bryn Mawr College in the procedure for defaced quartz.

Fig. 4 is from W. L. Bond, *Bell Syst. Tech. Jour.*, **22,** 224 (1943). Fig. 8 was obtained from Reeves Sound Laboratories, Inc., New York City. Dr. O. Ivan Lee of William B. Ogush, Inc., New York City, contributed information on the dop method and Fig. 9.

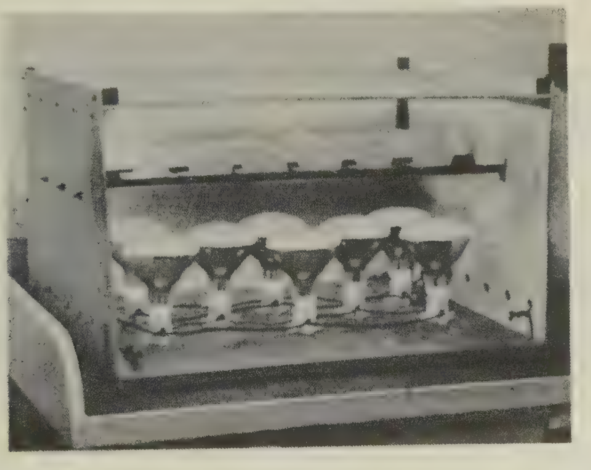

FIG. 3. Infrared lamp oven used for setting thermoplastic cement in mounting crystals.

MOUNTING QUARTZ FOR CUTTING

Some of the difficulty in the earlier period of crystal cutting was due to unsatisfactory cementing materials, which included plaster-of-paris, shellac, optical pitch, "iron glue," sauereisen, mixtures of plaster-of-paris, rosin and wax, water glass and whiting, and other substances. An ideal cement proved to be a thermoplastic.[4] This cement may be thinned with acetone and dispensed from a small oil can directly to a piece of plate glass for mounting. The cement becomes tacky after standing about ten minutes in air. The quartz is placed on the cement, and Z set parallel to the reference edge of the glass plate with the mounting stauroscope. Infrared lamps[5] are used to set the cement at a temperature of approximately 250° F. (Fig. 3).

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[4] General Electric Co., Bridgeport, Conn. Thermoplastic cement, ZV5057.

[5] General Electric, Wabash, Westinghouse, 115 V 250 W, reflector heat lamps with internal metallic reflectors.

Crystals have been mounted on various sorts of material for sawing. These included plaster blocks, plywood, masonite, metal and glass. The most satisfactory material is $\frac{1}{4}''$ thick plate glass cut into 3″ by 5″ rectangles. A straight reference edge on one long side is quickly ground on a large lap. The advantages of glass are its (a) rigidity, (b) will not damage diamond blade, (c) cheapness.

Single Mount Cutting Schemes for Small Quartz

The five methods described below are typical of the many schemes for cutting quartz by a single mount "once-for-all" method. In most of these methods, the orientation is carried out with the aid of an electrical polarity determination of X and the hand of the crystal in a polariscope. Since left-hand crystals had to be turned on the saws in the opposite direction from the right-hand crystals, various methods were devised to avoid confusion. In some plants a large R or L was painted on each saw, and it received quartz of only one hand.[6]

Flat Lay Cutting. One of the oldest cutting schemes is to mount the crystal on a prism face, the mount placed on a jig and transferred to a conoscope where the optic axis is made horizontal. The mount is transferred to the saw platform whose vertical angle has been preset to the desired wafering angle. The blade position is fixed and the crystal wafered by cross-cutting (Fig. 4). Larger crystals were first cut into Z-sections, the sense of the cut and Y-axis determined by *x*-rays, and the section wafered in the same manner.

The great disadvantage of this method is that the saw enters a slanting surface (the prism edge is at an angle to the blade) so that the blade drifts. It is therefore necessary to cut the wafers very thick (0.090″ thick compared with 0.045″ in the X-block method) and true each wafer by cementing on rather expensive specially designed cylindrical jigs which are corrected by *x*-rays and the wafers trued on an ultralap.

Direct Wafering. This method is also called "*baloney slicing*" in the industry. The crystal is sometimes held in a clamp and tilted up until the plane of the cut is parallel to the saw blade (Fig. 5). More often the crystal is mounted on a prism edge with the X-axis vertical and the plane

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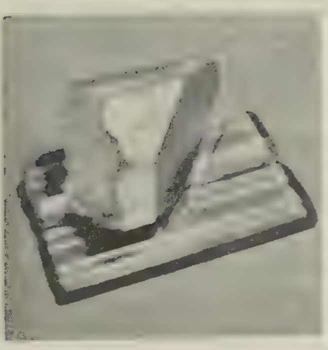
[6] When *x*-ray checks were introduced, a visitor might have been justified in thinking that a political revolution was imminent as sawyers thrust quartz wafers at the *x*-ray operator, crying "I am a left" or "I am a right." Elsewhere the operators of saws cutting left quartz were distinguished from those cutting right quartz by differently colored—but vivid and unmistakable—smocks. In still another plant, where the girls got careless in setting up quartz marked simply R or L, a psychological, if sentimental, approach was tried with great success: green paper hearts were pasted on left quartz crystals, and red paper hearts on the right crystals.

(4)

(5)



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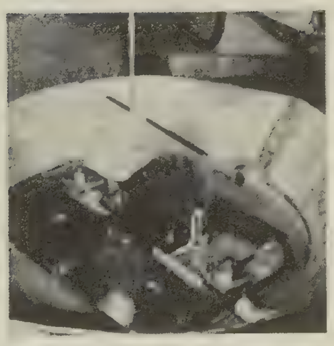

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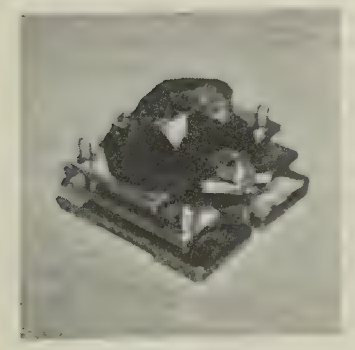

(8)

(9)

FIGS. 4–9. Single mount cutting schemes. (4) Flat lay cutting (Bond, *Bell Syst. Tech. Jour.*, **22,** 224, 1943). (5) Direct wafering. (6) Direct wafering by mounting crystal on prism edge in 120°V groove in glass plate. (7) Vertical glass mount. (8) Transfer jig. (9) Dop method.

of the cut made parallel to a reference edge of a masonite or plywood board. The orientation is accomplished by setting the crystal in plaster-of-paris or thermoplastic cement mixed with sand, and pushed around until the prism striations are made vertical with the aid of a right-angle square. Sometimes 120° V's are cut in wood or glass (Fig. 6). The difficulty of mounting the crystal with X vertical was later solved by simply grinding away a prism edge, or cutting it off.[7] This should not be confused with the precise cutting of a pair of such planes in the X-block method, for the latter is but one step in the inspection and reorientation of the crystal. The method requires identification of the major rhombohedron or determination of hand and polarity.

Vertical Glass Mount. The difficulty of setting up a quartz crystal with the X-axis vertical was overcome in the Chicago area by a novel scheme. The crystal is cemented on a prism face to a rectangular glass plate, with Z nearly parallel to the long reference edge. The divergence of Z from the reference edge was determined by a crossed Polaroid stauroscope using a photoelectric cell. Since the prism faces are irregularly developed, and therefore not parallel to Z, the divergence between Z and the plane of the glass plate on which the crystal is mounted was also found by turning the mount 90° on the stage. Being mechanically, rather than geometrically minded, the divergences were measured by feeler gauges rather than on a divided circle, and adjustments were made at the saw by similar coordinated gauges. The novelty of the method is that the glass plate is locked in the saw on its long edge (Fig. 7), i.e., with the plane of the glass perpendicular to the saw table. The blade cuts through the upper edge of the glass plate and then into the crystal.

Transfer Jig Method. The following description is typical of the schemes involving the use of a jig with more or less universal movements which is used for preorientation of the crystal by optical and/or x-ray methods and transferred to the saw for direct wafering. An approximate X-plane $(11\bar{2}0)$ is cut on one side of the crystal and one or both ends, perpendicular to Z, are also cut off to facilitate conoscope observations. The crystal is mounted with stick shellac on the X-plane on a circular fiber board which is clamped to the jig (Fig. 8). The jig is set on the track in the conoscope tank and the optic axis centered by a pair of adjusting screws and the hand determined. The jig is taken to a dark room and mounted in a special x-ray stand and the X-axis tilted to the vertical by adjusting another pair of screws until a pair of diffracted x-ray spots are centered on a fluorescent screen.[8] The jig is locked on the saw table which is set at the cutting angle and wafered. Test cuts are corrected by further x-ray checks.

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[7] See for example, Johnson, C. E., Method of cutting quartz: *U. S. Patent Off.*, No. 2,264,698, Dec. 2, 1941.

[8] The fluorescent screen method is used by few plants. In most cases the X-axis is oriented by triangular etch light figures on preliminary cut Z-planes or by x-rays using a Geiger counter tube.

Dop Method. This is a scheme in which faced crystals are preset to the cutting angle and wafered directly. The BT-cut is 10°47′ (for $ZZ'=49°$) from the major rhombohedron r. This rhombohedron plane is carefully ground by hand until a precision protractor indicates that the plane angle has been reduced 10°47′, which requires craftsmanship. The ground plane is cemented with glyptal resin to a steel block held by a vise on a standard milling machine (Fig. 9). The crystal is fed into the saw and successive cross-cuts are made parallel to the original ground surface. There is no correction of the sawing angle and off-angle wafers must later be corrected by lapping.[9]

THE X-BLOCK, WAFERING PROCEDURE FOR SMALL QUARTZ

This is the most widely used and successful scheme for cutting quartz weighing up to 1000 gms. Larger quartz is first cut into thick Z-sections and then into X-blocks, or if it contains large flawless areas, into thin Z-sections and then Y-bars, as summarized in Table 1. The procedures have been carefully studied and are given in sequence below. Orientation details are described in the preceding papers by the writers. Figures 10 to 15 illustrate the more important steps in the manufacturing scheme.

TABLE 1. SCHEMES FOR PROCESSING VARIOUS TYPES OF RAW QUARTZ USING THE X-BLOCK, WAFERING PROCEDURE

Size (grams)	Faced or Defaced	Cutting Scheme
<100	Faced and Defaced	Not economically feasible to cut at this time.
100–1000	Faced	X-block, wafering method.
	Defaced	Cut Z-planes and remount for X-block, wafering method.
1000–2000	Faced	If quartz contains large flawless portions use Z-section, Y-bar method. Otherwise thick Z-section, X-block method.
	Defaced	Grind mounting face parallel to Z and cut as faced quartz.
>2000	Faced and Defaced	Cut into smaller pieces which can be handled as described in weight classes above.

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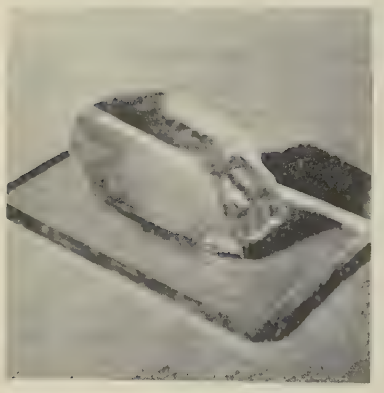
[9] The method is useful in salvaging ends which have fallen off the glass mount and already have the wafering plane on one side.

(10)

(11)



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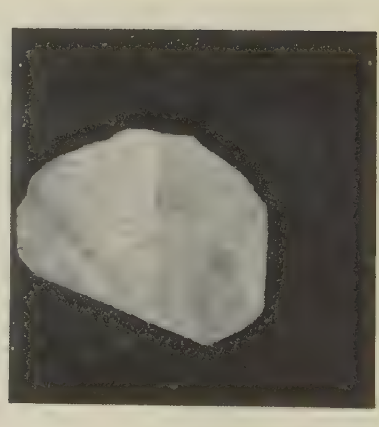

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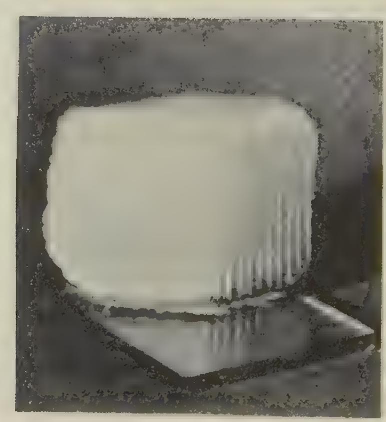

(15)

The crystal is mounted on a natural prism or apex face and X-planes $(11\bar{2}0)$ cut on both sides. The X-block is etched to permit inspection, maximum utilization of electrical twinned areas and to permit orientation by the parallelogram light figure thus eliminating polarity and hand determinations. The crystal is remounted on one of the X-planes and rotated to the desired wafering angle. Since the diamond blade enters perpendicular to the flat surface of the X-plane, and no mounting jigs are used, thus assuring a stable mount, the maximum sawing accuracy is obtainable. Rules for the coordination of saws, *x*-ray goniometer and optical devices are summarized in Table 2.

1. *Inspection and Grading of Raw Quartz.* This subject is covered in detail in another paper.[10] The inspectoscope is used to grade crystals and locate flaws before cutting. Optical twinning and physical imperfections such as bubbles, needles, etc., are found in this way.

It is good practice to sand blast and etch all crystals in concentrated ammonium bifluoride solution at room temperature for 10 to 20 hours before cutting so that the electrical twinning which is so prevalent can be identified. Sand blasting reduces etching time but large masses such as raw crystals and X-blocks should not be placed in hot etch solution which may crack them. Although hydrofluoric acid is better for this purpose than ammonium bifluoride solution because it produces a coarser and deeper etch, it is not recommended because of the industrial hazards involved. It is not always possible to determine the depth and position of all the electrical twinning in a crystal before cutting, but careful inspection has proven useful.

2. *Selection of Mounting Face.* The crystal may be mounted on any prism or rhombohedron face which is large and flat enough to properly support the crystal (Figs. 10 and 11) in cementing and cutting, and a line parallel to the optic axis is marked on the mounting face. The greatest yield will be realized by selecting a mounting face on the side of the crystal which shows no evidence of electrical twinning. The electrical twin then appears on the etched surfaces of the X-block (Fig. 13) which could be cut along the twin boundary and each side wafered as an individual crystal. If the electrical twin is mounted on the twin boundary the twin plane will be concealed in the X-block and the pin-hole light figure will be the same on both sides. It is then impossible to determine how the block should be oriented for wafering because of the difficulty in tracing the twin boundary.

The optical twin laminae may be parallel to any one or all three major rhombohedron faces. If optical twinning is observed in the inspectoscope to consist of a series of parallel bands extending across the crystal, a greater yield will be obtained by mounting on that face showing the most twin laminae. When the crystal is remounted on the X-plane, the BT-cuts which are approximately parallel to the major rhombohedron can be made be-

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[10] Gordon, Samuel G., The inspection and grading of quartz: *Am. Mineral.*, this issue; The inspection of quartz, *Manual for the Manufacture of Quartz Oscillator Blanks, Supp. No. 2*, Office of the Chief Signal Officer, War Dept., Washington, D. C., July 1, 1943.

FIGS. 10–15. X-block, Wafering Method. Crystal is mounted on prism (10) or rhomb (11). X-planes cut to form an X-block (12). (13) Electrical twin may be cut along twin boundary and each part individually wafered. (14) X-block remounted for wafering and flawed portions painted out to avoid useless sawing. (15) Wafer "comb."

tween rather than across the twin laminae (Fig. 16). The rule fails for AT-crystals which are approximately parallel to the minor rhombohedron for then one cannot help cutting across the twin laminae. If the optical twinning is close to one side of the crystal, it is better to mount it so that side is cut away in cutting the X-planes.

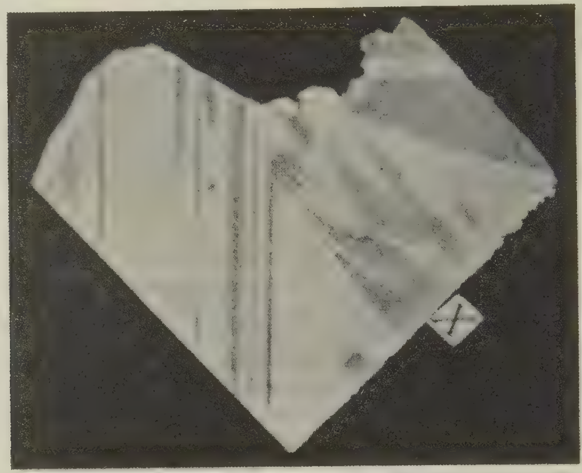

FIG. 16. This wafer as cut will yield no blanks. If the quartz had been set up with the optical twin laminae vertical (for these are parallel to another X-axis), it would have been possible to cut some usable wafers from between the twin laminae.

3. *Mounting for Cutting X-Planes.* The crystal is mounted with its optic axis parallel to the long reference edge of a piece of thick plate glass (usual size, 3″×5″) with thermoplastic cement. After the cement becomes tacky the mount is transferred to the stage of a mounting stauroscope and the reference edge of the glass placed flush against the reference edge on the stage. The latter has been set parallel to the plane of polarization of one of the Polaroids. The crystal is shifted to the extinction position so that the optic axis of the crystal is now closely parallel to the reference edge of the glass plate. The mount is then transferred to an infrared lamp oven and baked about an hour at 250°F. to harden the thermoplastic, and allowed to gradually cool to room temperature.

4. *Cutting X-Plane.* The mount is placed on the saw with the reference edge of the glass plate flush against the reference edge of the saw table which is parallel to the saw blade (saw table scales read 0°0′ for both horizontal and vertical angle). A vertical arrow pointing up is marked on the outer surface of the crystal (side opposite the fresh cut side) and a thin test piece is cut from either side of the crystal. The blade must not be forced; the technique of sawing is described in detail in an accompanying paper.[11]

5. *X-Ray Measurement of X-Plane.* Burrs must be removed from the fresh cut surface of the test piece to prevent errors in x-ray measurement. The x-ray goniometer is set with a standard to 0° for reflection from $(11\bar{2}0)$, $2\theta=36°34'$ for Cu$K\alpha$.[12] The test piece is placed

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[11] Parrish, William, Methods and equipment for sawing quartz crystals: *Am. Mineral.*, this issue.

[12] Parrish, William and Gordon, Samuel G., Precise angular control of quartz cutting with x-rays: *Am. Mineral.*, this issue.

in the x-ray crystal holder with the fresh cut side toward the x-ray beam and arrow facing the operator. The arrow is pointed up for measuring the horizontal angle and to the left (if cut from the left side of the crystal) or the right (if cut from the right side of the crystal) to measure the vertical angle. The saw table must be corrected by the *same amount and direction* as indicated by the direct x-ray goniometer reading. Steps 4 and 5 are repeated until an X-plane within the tolerance limits is obtained. With proper equipment and some care only one correction is required to bring the cut to a tolerance of ±15′ to 30′.

6. *Cutting Second X-Plane.* A second X-plane is cut on the other side of the crystal to make an X-block (Fig. 12). The procedure is the same as in steps 4 and 5.

7. *Etching of X-Block.* The X-block is removed from the glass plate by immersing the mount for an hour in acetone. The crystal is cleaned and etched as described in step 1.

8. *Inspection of Etched X-Block.* The block is inspected in a spotlight for both optical and electrical twinning. Usable electrical twinned areas are marked along the twin bound-

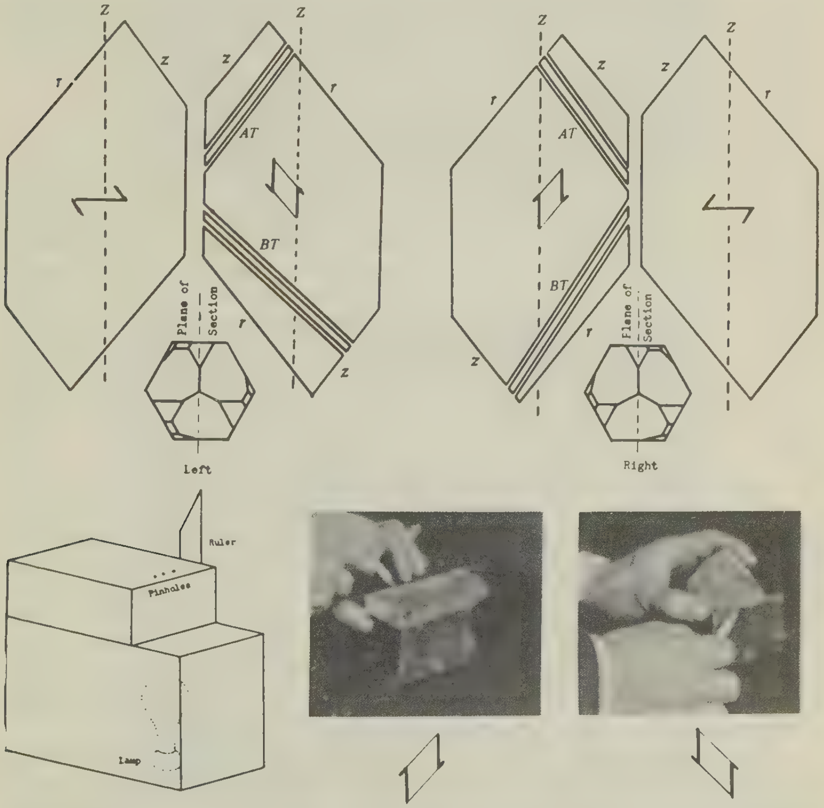

FIG. 17. Orientation with parallelogram light figure. The four possible light figures on X-sections are shown above. The rule is to turn to the side showing the parallelogram and rule a line parallel to Z (the short side of the parallelogram) on the top side if r slopes NE-SW, or on the under side if r slopes NW-SE. The ruled side is mounted down so that in all X-blocks r slopes NW-SE, thus simplifying saw table settings and correlations with x-ray goniometer readings.

ary for cutting apart and processing each part separately. To avoid processing of those parts which will give no yield, badly twinned and flawed areas may be marked out (Fig. 14).

9. *Orientation of Etched X-Block.* The etched X-block is placed on a pin-hole light box with the face showing the parallelogram light figure up. The crystal is rotated in the horizontal plane until the optic axis direction, the short side of the parallelogram, is schematically north-south. If the long side of the parallelogram, the major rhombohedron direction, slopes NE-SW, a line parallel to the optic axis is ruled on the top side of the X-block. If the long side slopes NW-SE, a line parallel to the optic axis is ruled on the under side of the X-block (Fig. 17).

10. *Mounting X-Block.* Same as step 3: ruled face must be in contact with glass.

11. *Orientation of X-Block on Saw Table.* The procedure outlined in steps 9 and 10 assures that the major rhombohedron of all X-blocks will slope in the same direction when mounted, regardless of the hand of the crystal or the polarity of the X-axis. The orientation of the X-block and correlation of the saw table and x-ray readings is thus simplified and an easily visualized set of rules (Table 2) can be applied for all types of cuts. With the long reference edge of the glass plate locked against the reference edge of the saw table, AT-type cuts are turned counterclockwise to the desired ZZ′ angle and BT-type cuts clockwise (Fig. 17).

12. *X-Ray Check of Test Cut Wafer.* A thin test cut is made from one side of the X-block, marked and checked by x-rays using exactly the same procedure as in Steps 3 and 4. The $(01\bar{1}1)$ atomic planes, $2\theta = 26°40'$ for Cu$K\alpha$ are used for AT-cuts and $(20\bar{2}3)$, $2\theta = 68°06'$ for BT-cuts. A sawing precision of the order of $\pm 10'$ to $15'$ is required for ZZ′ and XX′, the horizontal and vertical angles, respectively, of the saw table. Since the surface of the wafer is inclined to the atomic reference planes, the horizontal and vertical corrections must be read from different points of the x-ray goniometer scale (XX′ $38°13' -$ ZZ′ $35°15' = 2°58'$ apart for AT-cuts and XX′ $49°44' -$ ZZ′ $49°20'$ (nominal) $= 24'$ apart for BT-cuts). Where large production is required, it is advisable to set up one goniometer to measure ZZ′ directly, and the other XX′. The correlation of x-ray readings and saw table orientation for AT- and BT-cuts for horizontal and vertical angles should be made in the manner described in steps 5 and 6 using a vertical arrow pointing up to preserve the direction.

13. *Wafering of X-Block.* When a perfectly aligned wafer is evinced by x-rays, the quartz is sliced to wafers of minimum thickness with proper allowance for quartz to be removed by lapping and etching. For 6 to 8 MC BT-crystals (0.0125″ to 0.0167″ final thickness) using a metal bonded diamond blade, such as the Norton 8″ diameter, 0.050″ thick, #80 grit-size, L25M concentration, mounted on a $3\frac{1}{2}$″ flange, the wafers may be safely cut 0.045″ thick (Fig. 15). Unless the ways of the saw table are accurate, each 3rd or 4th wafer should be checked by x-rays to make certain the cuts are within angular tolerance.

14. *Etching of Wafers.* Wafers are cleaned with a solvent such as carbon tetrachloride, set vertically in slotted trays (Fig. 18) and placed in a warm (50°C.) solution of concentrated ammonium bifluoride for $1\frac{1}{2}$ to $2\frac{1}{2}$ hours. A good etch is required.

15. *Inspection of Etched Wafers.* The wafers are examined with a spotlight or twinoscope for optical and electrical twinning, cracks, etc., and all flawed areas are marked out. The usable portion of electrically twinned wafers may be determined by the use of any one of several techniques: (a) position of the twinned portions with respect to the X-planes reference edges which show how the block was oriented with respect to the parallelogram; (b) by differences in transparency of electrically twinned etched portions (only *one* portion of the electrical twin is usable—the more transparent side is nearly parallel to r and usable in BT-wafers; the more opaque side is nearly parallel to z and usable in AT-wafers); (c) by

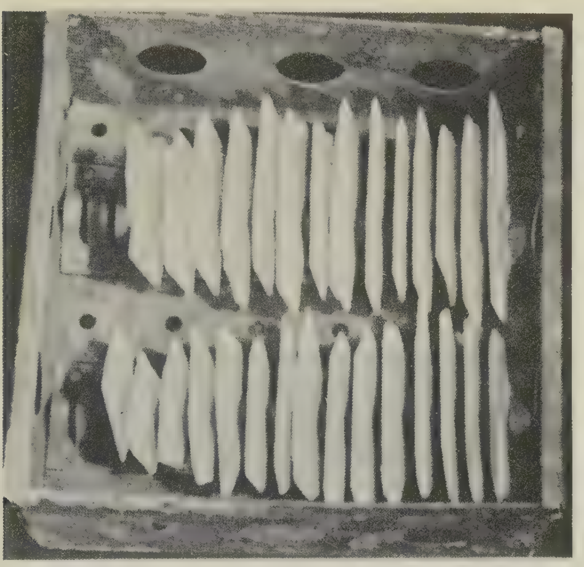

FIG. 18. Wafers are placed in copper trays with notched bases for etching.

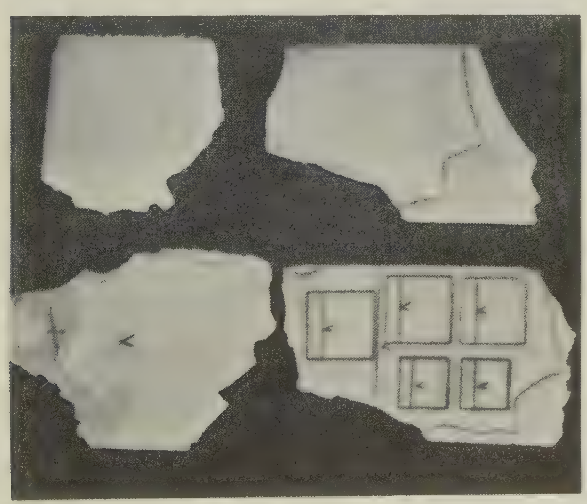

FIG. 19. Procedure for wafers. After etching, the twinned and flawed areas are marked, the usable portion of electrical twins determined, stauroscoped for X and Z, and finally laid out with a rubber stamp. Small blanks are laid out in those areas not large enough to yield the larger blanks.

differences in light reflection; (d) by an x-ray test using atomic planes which have different reflection intensities on opposite sides of the optic axis such as $(01\bar{1}1)$ and $(10\bar{1}1)$ for AT-cuts and (2023) and $(02\bar{2}3)$ for BT-cuts (the side with the weaker reflection is the usable side in both cases); or by a force oscillator and thickness measurement to determine the approximate frequency-thickness constant.

16. *Lay-Out Wafers*. The X and Z directions are determined and marked on each wafer with the angular view stauroscope. X is perpendicular to the two reference edges on the wafer remaining from the X-block stage (Fig. 19). It is therefore only necessary to determine the direction of the optic axis with respect to the plane of the wafer and precision is not required. The unflawed portions of each wafer are then layed out with a rubber stamp (Fig. 20) which is slightly larger (say 0.035″) than the finished blank and has the X and Z directions indicated. It is not necessary to precisely orient the rubber stamp with respect to the reference edges but the X and Z directions determined in the stauroscope are retained.

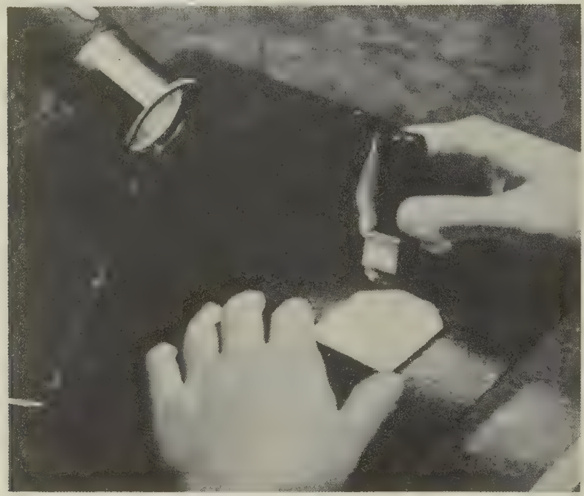

Fig. 20. Lay-out of wafer with rubber stamp.

17. *Trim Sawing of Wafers*. The trim saw is set up with its side reference edge exactly the same distance from the blade as the desired width of the cut blank. By placing the reference edge of the wafer against the back reference edge of the trim saw table, the dicing angle is maintained with respect to the X-axis.

18. *Edging of Blanks*. There are several methods for edging and squaring blanks to final dimensions. They may be loafed together and squared by a machine lap or individually squared with a metal bonded diamond abrasive wheel.[13]

19. *Inspection of Blanks*. All blanks are x-rayed for ZZ′ and XX′ angles, inspected for squareness and flaws with the aid of a spotlight to insure only perfect blanks being sent to the lapping machines.

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[13] Parrish, William, *op. cit.*

20. *Machine Lapping of Blanks.* The entire high speed lapping to frequency technique is described in an accompanying paper [14] Three stages using successively finer abrasives, finishing with #303 aluminum oxide are used to bring the blanks within 15 to 30 KC of final frequency. The blanks are then inspected and classified for frequency for final frequency adjustment.

21. *Final Finishing of Blanks.* The blanks are now ready for etching-to-frequency. Usually a two stage etching process is employed: mass etching to within a few KC of final frequency followed by a slow etch to the final point.[15]

Coordination of Saws, X-Ray Apparatus and Orientation Devices. It is manifest that absolute coordination of the orientation devices with the saws and *x*-ray measurements is necessary. It is possible to do this with a few simple rules and without bothering about the hand of the quartz, or the polarity of its axes, or whether *x*-ray goniometers are used with circles reading clockwise or counterclockwise.

Saws. For precise cutting it is necessary that the saw cuts true; that the plane of the blade is perpendicular to the plane of the saw-table, and that coolants are used that work efficiently and prevent warping of the blade and heating of the quartz. A reference bar is fastened to the saw table, which should be in perfect alignment with the saw blade when the circle reads 0°.

X-Ray Equipment. Standards should be cut from quartz crystals and used to set the goniometer scale to 0° for each atomic plane used.

Mounting Stauroscope. This can be adjusted by means of a piece of Polaroid whose plane of vibration is parallel to an edge. This edge is set against a reference bar on the stage of the instrument. The lower Polaroid is turned to extinction. The test Polaroid is removed, and the analyzer is turned to extinction.

Angular View Stauroscope. The test Polaroid is set against the ruling guide, and the polarizer is brought to extinction, and after the test Polaroid is removed, the analyzer is brought to extinction.

Pin-hole Light Box. The rulers can be set by means of the parallelogram shown by an X-block.

PROCEDURE FOR SMALL DEFACED QUARTZ

A few additional steps are required to process a defaced crystal. Some of the cumbersome methods used in the crystal industry were developed in order to process all raw crystals, faced and defaced, the same way. Al-

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[14] Parrish, William, Machine lapping of quartz oscillator-plates: *Am. Mineral.*, this issue.

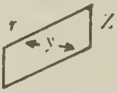
[15] Frondel, Clifford, Final frequency adjustment of quartz oscillator-plates: *Am. Mineral.*, this issue.

TABLE 2. RULES FOR COORDINATION OF SAWS, *X*-RAY GONIOMETER AND ORIENTATION DEVICES

MOUNTING. Quartz is mounted on prism or rhombohedron with Z parallel to long reference edge of glass plate, using mounting stauroscope.

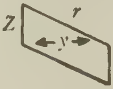
PIN HOLE LIGHT FIGURE.

Major Rhomb Slopes NE-SW



Rule Z on top side
of X-Block

Major Rhomb Slopes NW-SE



Rule Z on under side
of X-Block

SAW TABLE TURNS

Cut	*Direction*	*Angle from 0°*
Z-Plane	*C* or *CC*	90°
X-Plane	—	0°
AT	Counterclockwise	35°15′
BT	Clockwise	49°20′

MARKING OF TEST CUTS AT SAW

Mark vertical arrow pointing up ↑ on outer surface (opposite fresh cut side) of all test cuts.

X-RAY SETTING OF TEST CUTS

Remove burrs and place test cut flush against crystal holder with fresh cut side toward *x*-ray beam and arrow in view of operator.

Facing Saw, Test Cut from	To measure horizontal angle (ZZ′) place arrow	To measure vertical angle (XX′), place arrow
RIGHT SIDE	↑	→
LEFT SIDE	↑	←

Set *x*-ray goniometer to read *directly* the angular divergence. For example, for ZZ′ of an AT-cut, set goniometer scale to 0°0′ for desired ZZ′ angle for maximum reflection from $(01\bar{1}1)$ $2\theta = 26°26'$. Rules apply to left or right goniometer using *C* and *CC* conventions.

CORRECTIONS AT SAW.

X-ray goniometer readings are *duplicated* at the saw. If a reading was *clockwise* from zero at *x*-ray, saw table is also turned *clockwise*, and vice versa.

ANGULAR-VIEW STAUROSCOPE.

Bring wafer to extinction, then

(1) Draw line left to right (X-direction) ——
(2) Mark arrow-head pointing away from observer at far side of blank (Z) —△—

X-RAY MEASUREMENT OF CUT WAFERS AND BLANKS.

For direct readings set *x*-ray scale to 0° for desired angle and orient as follows,

Cut	ZZ′	XX′
AT	◁|	—△— or —▽—
BT	|▷	—△— or —▽—

though this is desirable, it is certainly a mistake to handicap a procedure by treating the large majority of the crystals by the more difficult methods required for the small minority of defaced crystals. It is therefore advisable to take advantage of the natural faces on the crystal and process all faced crystals by the X-block method described above and to set up a special procedure whereby defaced crystals are made into "faced" ones and *then* all crystals can be handled in the same manner.

The cutting of defaced quartz presents no special orientation problems and involves only the cutting of a pair of Z-planes and remounting the crystal for cutting X-planes. The great difficulty in the past has been the mounting of large, quite irregular masses—with curved, slippery and sharp surfaces. Radio engineers have devised elaborate gimbals with tangential screws, and bicycle chains and vises. In one large plant the quartz was pushed around in a conoscope while embedded in modeling clay; it was then circled by a paper mold into which hot wax was poured. The problem was finally solved with the very simple suggestion:[16] "Why not grind a small surface on the crystal so that it could be cemented to a glass plate?" In this process, the optic axis direction is located by means of the conoscope, and the quartz is lifted out and without altering its horizontality is pressed to a rotating lap for a few minutes, sufficient time to grind a surface of about 1″ square, suitable for cementing the mass to a glass plate.

A simple jig which allows rocking the crystal in the vertical plane is used only for cutting the Z-planes in order to avoid excessive tilting of the saw table in this operation. This jig based on a design by W. L. Bond and shown in Fig. 21, permits the glass mounted crystal to be locked on the tilting platform whose position can be varied and locked by means of a pair of screws on opposite corners. A track on the under surface fits flush over the track on the bottom of the conoscope tank so that the horizontal correction for Z can be read directly from the conoscope scale. About 40 to 50 defaced crystals can be converted into "faced" crystals, mounted and ready for the X-block procedure by a few people using one saw in a normal working day.[17]

1. *Preparation of Mounting Surface.* The defaced crystal is turned in a conoscope until the optic axis is in view. The crystal is lifted out, keeping the optic axis direction horizontal, and pressed on a large rotary lap until a surface large enough for mounting purposes (about 1″ sq.) is ground. The crystal is returned to the conoscope, setting its ground surface on the flat surface on the bottom of the tank, and checked to make sure it is within about 10° of parallelism with Z. A line is drawn parallel to Z on the ground surface.

2. *Mounting for Cutting Z-Planes.* Using the mounting stauroscope, the crystal is mounted on a glass plate with Z parallel to the long reference edge.

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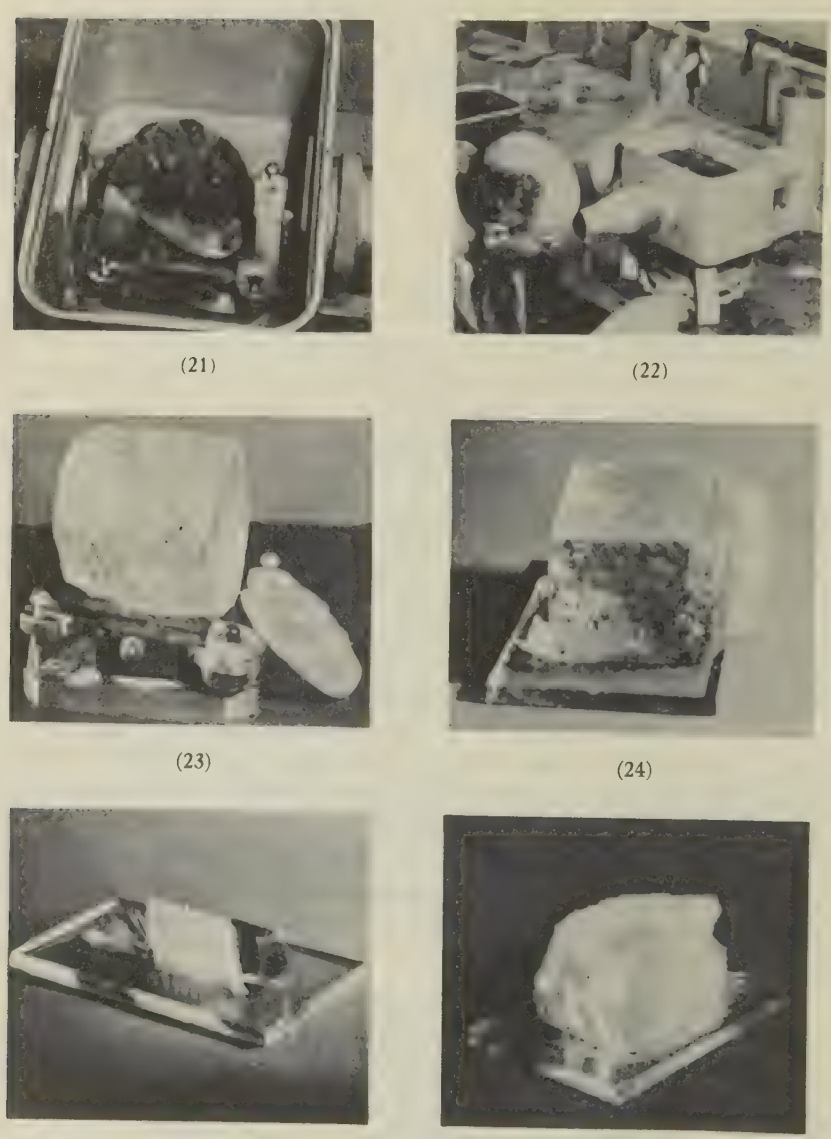
[16] This suggestion, so reminiscent of Columbus and the egg, was made by Miss Judith Weiss (now at Bryn Mawr College), then engaged in setting up the cutting procedure in the crystal shop of the Philadelphia Signal Depot.

[17] Using the procedure described here, nearly a ton of defaced quartz (over 2200 raw crystals) was prepared by a few people using one Felker #120 saw in less than two months.



(21) (22)

(23) (24)

(25) (26)

Figs. 21–26. Procedures for large and defaced quartz. (21) Defaced quartz mounted on jig in conoscope tank. (22) Adjustment of vertical angle of jig in conoscope. (23) Thick Z-sections cut from large defaced quartz. (24) Thick Z-section cut into an X-block; when remounted it will be ready for wafering. (25) Cutting blanks from a thin Y-bar; flawed portions painted black to avoid useless cutting. (26) Six Y-bars cemented together for wafering. Sides covered with glass to avoid chipping sides. This is nearest successful approach to "gang" cutting yet attempted.

3. *Orientation on Jig.* The mounted crystal is locked on the conoscope jig, placed in the conoscope so that its reference track rides on the track in the conoscope and the screws are adjusted until the optic axis is on the horizontal cross-hairs (Fig. 22). The horizontal deviation from zero and its direction are read on the conoscope circle and marked on the crystal.

4. *Cutting Z-Plane.* The jig is locked on the saw table with its reference edge flush with the reference edge of the saw table, the latter corrected in the horizontal plane as indicated by the conoscope reading, and a test cut is made perpendicular to the optic axis. An arrow pointed upward is marked on the outer surface of the test cut.

5. *X-Ray Measurement of Z-Plane.* The test cut is placed in the x-ray crystal holder with arrow pointing up and measured for horizontal and vertical angle corrections in exactly the same manner described above in the X-block method, step 5. The (0003) atomic planes are used; $2\theta = 50°40'$ for $CuK\alpha$. Steps 4 and 5 are repeated until a Z-plane within tolerance (30′ to 1°) is obtained.

6. *Cutting Second Z-Plane.* A second Z-plane is cut on the other side of the crystal and checked by x-rays using the method outlined in step 6 of the X-block procedure.

7. *Etching of Crystal.* The crystal is removed from the glass plate by immersing the mount for an hour in acetone. The crystal is cleaned and etched as described in step 1 of the X-block method.

8. *Orientation of Etched Crystal.* The pin-hole light figure shows three brightest spots forming an equilateral triangle with sides parallel to the Y-axes. A Y-direction is marked on the crystal which will yield the largest X-block.

9. *Mounting for Cutting X-Planes.* The crystal is mounted on a Z-plane with the chosen Y direction parallel to the long reference edge of the glass plate.

The crystal is now ready for processing by the X-block method starting with step 4. The only difference in the two procedures is the optic axis is perpendicular to the glass plate but in the X-block method it is approximately parallel to the glass plate. This, however, causes no change in procedure.

Cutting Schemes for Large Quartz

Single mount direct wafering methods are not feasible for quartz weighing more than 1000 gms. because (a) enormous waste caused by electrical twinning, (b) lack of accuracy in wafering due to excessive blade drift, (c) enormous increase in time required for cutting large wafers. Instead, the practice is to cut the quartz into sections, and then into bars which can be reduced to blanks. The sections are most efficiently and commonly cut perpendicular to the optic axis (Z-sections) because of the ease of locating Z in the conoscope, a choice of three cutting directions for the bars (Y-axis) is available to avoid flawed and twinned areas, and the cut is most likely to be across the shortest section of the crystal thus minimizing errors due to saw drift. The thickness of the sections is determined by the quality of the quartz, size of available diamond blade and size of final blanks. The sectioning methods require three different orientation, inspection, cementing and cutting steps going successively from larger to smaller pieces. The maximum yield and accuracy is obtained in cutting blanks from bars because thinner and smaller blades can be used. Success in cutting large quartz is proportional to the precision with which the first cuts are made and the procedure should permit checks of cutting accuracy and methods of correcting errors.[18] Large unfaced crystals may be treated preliminarily by the same method described above for mounting and cutting Z-planes. If the crystal is very large it is cut into smaller pieces preferably parallel or perpendicular to Z.

Z-Section, Y-Bar Method. The most efficient method for cutting large quartz of exceptional quality is that of cutting Z-sections about the same thickness as the width of the desired blanks (Fig. 27). The crystal is mounted on a prism face with thermoplastic cement on a glass plate. If the prism face is excessively inclined to Z the mount may be placed on a jig of the same type as described above for cutting unfaced quartz, or a

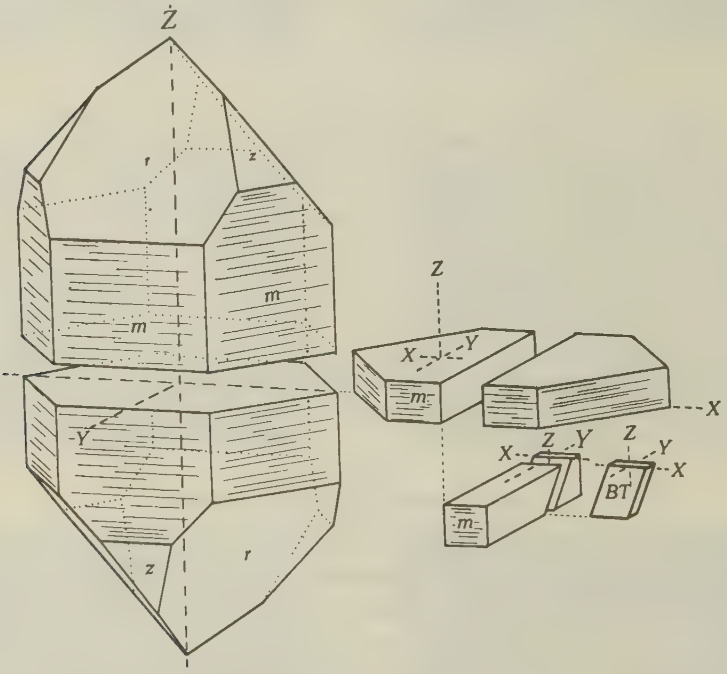


FIG. 27. Thin Z-section, Y-bar method is used for large quartz containing large flawless areas.

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[18] In some plants little effort was made to cut the first planes accurately. It was believed that such errors could be corrected later in the procedure. The failure to appreciate the necessity of starting with a precisely oriented plane was inherited from habits acquired in making X-cuts in the earlier days of radio, when a large number of quartz crystals were set in plaster-of-paris in rows and even piled on top of one another to be fed simultaneously into a 24″ saw on an adapted milling machine, or were cut up on gang "muck" (loose carborundum) saws.

more nearly parallel face ground on the crystal. The sections may be cut to an accuracy of ±15′ to 30′ and checked by x-rays using (0003) planes or with the conoscope. The Z-sections are then etched about 10 hours in concentrated ammonium bifluoride solution at room temperature, inspected and twinned and flawed areas marked out, and the Y-direction identified from the prism faces, by means of the pin-hole light figure, by x-rays or by the thermal parting method. The Y-bars are also cut the same thickness as the width of the desired blanks to an accuracy of ±15′ to 30′ using $(11\bar{2}0)$ planes for x-ray checks of test cuts. The bars are etched, oriented by the pin-hole parallelogram light figure, electrical twinned portions marked for cutting apart, twinned and flawed areas painted black to prevent useless sawing, and remounted for final slicing into blanks (Fig. 25). In cases where real precision has been achieved in cutting the bars, a number of them may be cemented together and wafered at the same time (Fig. 26). This is the nearest approach to gang precision cutting that has been attempted with any degree of success, and is done with a single blade.

Thick Z-Section, X-Block Method. When the quartz is not of the best quality, a modification of the above method will give a greater yield. The Z-sections are cut thicker and these are cut parallel to a Y-axis direction into X-blocks (Figs. 23 and 24). The X-blocks are then wafered and blanks can be diced from clean, untwinned areas of the wafers. In quartz of average radio quality, this method is much more flexible than the one described above, for it permits some selectivity in the cutting strategy.

X-Section, Complementary Bar Method. This method is an inheritance from the X-cut crystal procedure. The quartz is mounted on a prism face and sawed into sections parallel to the YZ plane (Fig. 28). Using the parallelogram light figure on an etched X-section, a direction which is the complement of the angle of cut is desired is ruled on the section (in the case of a BT-cut, this would be $90° - 49°$ or $+41°$ to the optic axis). The X-section is then cut up into complementary bars, from which blanks can be sliced at right angles to the elongation of the bars. The disadvantage of this procedure was the difficulty of cutting large X-slabs with accuracy, and it was difficult to check the accuracy of the cut and to true it, which were advantages presented by the Z-section methods.

Complementary BT-Section Method. This method was evolved in one plant to provide sections which could be wafered on a gang saw. A large quartz crystal was oriented successively in a conoscope and by x-rays and a universal transfer jig was used between conoscope, x-ray and saw. Mounted upon a prism face, the crystal was tilted to the cutting angle and complementary BT sections (0.6″ thick) were cut at the complement of the BT angle. The complementary BT sections were then slivered on a gang saw of 5 six-inch blades. Flanges were wide, so that only 0.7″ of the blades were available for cutting—a practical limit. The coolant was fed through the shaft of the milling machine, and out through the flanges and spacers between the saw blades.

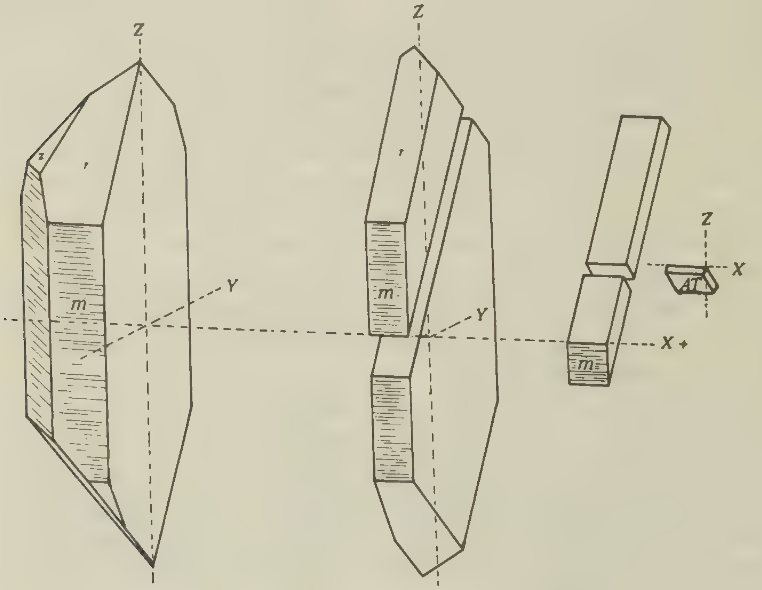

FIG. 28. X-section, complementary bar method.

Complementary Cylinders. A large quartz crystal mounted on a prism face, is tilted to the complementary angle of the cut desired. By means of a tubular drill of the desired diameter, set with diamonds on its cutting edge, cylinders of quartz are cut from the crystal. The cylinders may then be sliced perpendicular to their elongation to yield circular blanks.

METHODS AND EQUIPMENT FOR SAWING QUARTZ CRYSTALS

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Abstract

The principal types of equipment and methods for sawing, dicing and squaring quartz crystals are described. In down-cutting, the most widely used method, the diamond blade moves downward into the crystal which is fixed. The Felker-type saw with work-table adjustable for horizontal and vertical angle corrections determined by x-ray measurements is the most successful. Surface grinders and milling machines are not as good because they require expensive reconversion, the quartz must be preset, and little or no angular correction is possible. Diamond blades of the notched or metal bonded type are used. Their efficiency and accuracy are dependent upon the quality of the sawing machine, type of entering surface, size of cut, speed, pressure and coolant. The best results are obtained when the arc of contact is small, the entering surface flat and perpendicular to the blade, the saw relatively free of vibration, large flanges and a copious flow of proper coolant, low pressure and relatively high blade speeds (4000–6000 s.f.m.). Methods for dicing and squaring are also described.

Introduction

The quartz crystal industry employs a huge number and varieties of sawing equipment and methods. Only a few of the most successful of these can be described here. The sawing equipment to a great extent determines the orientation procedures and vice versa. Cutting schemes and orientation techniques are described in detail in previous papers.[1]

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Both equipment and procedures admittedly could be considerably improved. In common with most things in the quartz industry they were hurriedly developed for the emergency. The job has been an amazing accomplishment when we realize that several hundred million *blanks* were cut in less than three years. Generally speaking, the best results are obtained in those plants which maintain a well-equipped shop for servicing the numerous mechanical details that are continuously required. Mineralogists will find many of the techniques useful and may obtain excellent detailed information on the operating conditions and equipment from the manufacturers and nearby crystal plants.

Acknowledgments. The crystal industry is indebted to the Felker Manufacturing Co., Inc., Torrance, Calif., manufacturers of saws and rim-lock diamond blades and the Norton Co., Worcester, Mass., manufacturers of metal bonded-type diamond blades for their cooperation in making available equipment for cutting quartz crystals. Some of the data included in the section on diamond blades were obtained from Mr. C. R. Van Riper, Research Laboratories, Norton Co. The photograph of the crystal edging machine, Fig. 14, was submitted by Volkel Bros. Machine Works, Los Angeles, Calif.

SAWING METHODS

The four principal methods of cutting quartz are shown in Fig. 1. The most widely used method for wafering and cutting blanks from bars is *down-cutting*. The crystal position is fixed and the blade moves into the crystal. In the Felker-type of saw, an automatically controlled variable hydraulic retardant, controls the rate of down-feed. This is superior to manual control or free-falling type of control in which the down-feed is as fast as the blade is cutting.

In *cross-cutting* or through-cutting, the vertical blade position is locked and the crystal is pushed, manually- or motor-driven into the blade. This is a useful procedure when the entering surface in down-cutting is at an angle to the blade and it is difficult to obtain a smooth cut (e.g., cutting X-sections on faced crystals). The cut is started at the broken end of the crystal for the apex end is just as difficult to enter as the sloping prism faces. The method is not widely used and has been more successful when a power-driven feed is employed.

Rotary and step cutting are commonly used abroad but not in this country. There is no machine available here for the former, and only an experimental model for the latter, and it has not been successful. The equipment required for this type of sawing is far more elaborate and expensive than that commonly used here. It has been stated that the advantage of these methods over those described above is that diamond surface is in contact with the quartz only over a very small arc and hence the blade is more efficient, cuts faster and with less saw marks. The little experimental work carried out thus far has not borne out these claims. In rotary cutting, the vertical blade position is fixed and the crystal

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[1] Gordon, Samuel G., and Parrish, William, Cutting schemes for quartz crystals: *Am. Mineral.*, this issue. Orientation techniques for the manufacture of quartz oscillator-crystals: *ibid.*

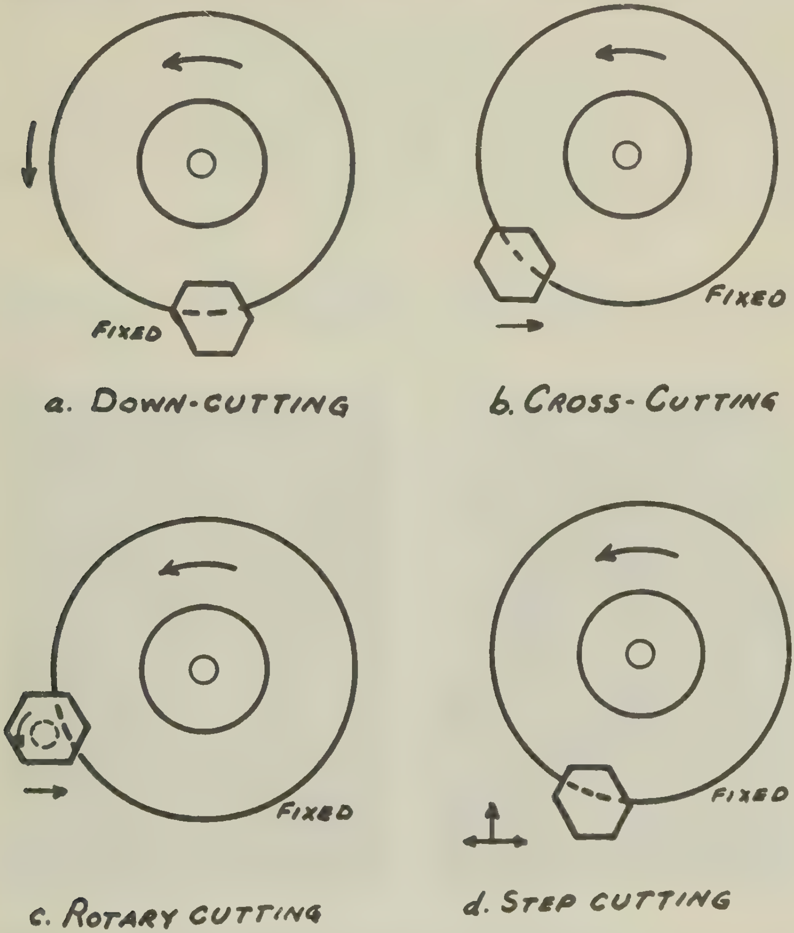
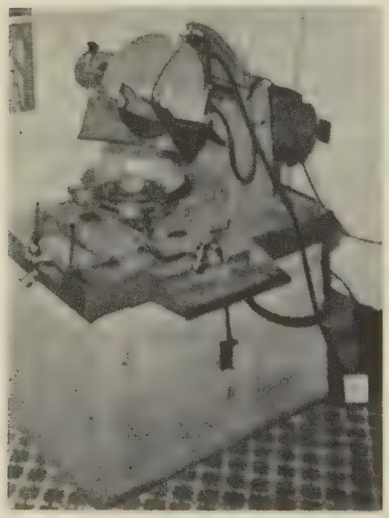


FIG. 1. Sawing methods. Down-cutting is the most widely used and successful method. The methods and equipment used in the diamond industry were found to be totally inadequate for quartz.

rotated in the same direction as the blade at low speeds up to 50 r.p.m. and moved into it. One of the disadvantages is that a small tip remains on the center of the wafer and must be lapped off. In addition, the wafer is tapered toward the center. In step cutting, the vertical blade position is also fixed but the crystal is mechanically driven back and forth under the blade and moved up slightly with each sweep. This is the method sometimes employed on surface grinders converted for quartz work. The experimental work conducted thus far shows that the wafers have relatively deep saw ridges, and the cutting is slower and more complicated than by the methods commonly employed.

SAWING EQUIPMENT

Two principal types of saws are used in the quartz crystal industry. The Felker-type[2] permits angular adjustments in the horizontal and



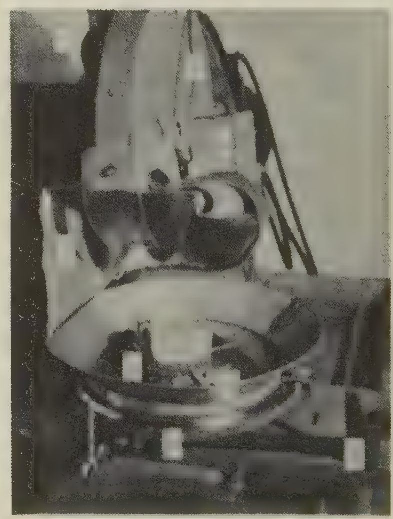


FIG. 2. Felker saw mounted on concrete block. (A) Adjustable counterweight; (B) depth stop; (C) coolant tank and circulating pump.

FIG. 3. Felker rotary table. (A) Hydraulic retardant; (B) pipe for feeding coolant into flange; (C) housing for light; (D) apron for splash guard; (E) reference edge; (F) clamps for holding mount; (G) graduated circle and vernier; (H) vertical angle scale.

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[2] Manufactured by Felker Manufacturing Co., Torrance, Calif. The most widely used are the model #80 and #120 (same as #80 but larger), with HVCT-12 rotary table. The Atlas Sales Co., Chicago, Ill., makes a quartz cutting saw converted from a bench model drill press.

vertical plane and has been the most successful and widely used type employed in this country (Figs. 2–3). The converted milling machine type of saw allows little or no angular adjustments and requires the quartz to be preset to the proper cutting angle (Fig. 4). Both types require considerable mechanical work before being placed in operation.

Felker Saw. The spindle carrying the blade and arbor are part of the same assembly and move in a radius about a pivotal point in the back of the saw (Figs. 2 and 3). A heavy adjustable counterbalance weight connected to a pin at the rear of the yoke permits varying the blade pressure. An adjustable hydraulic retardant controls the rate of down-feed. The rotary table and pulley must be carefully covered to prevent abrasive action of the quartz particles. The horizontal angle scale is graduated to degrees around the entire circumference and a vernier permits reading to 1′. The vertical angle is marked at 10′ intervals and can be tilted 10° on either side of the horizontal. Two manually operated screws, each graduated to 0.001″, permit translating the crystal parallel and perpendicular to the blade. The table requires the installation of a flat plate with reference edge adjustable to parallelism with the blade when the scale is set to 0°0′. This plate also should contain three slots, 90° apart, for screws and clamps for holding the mounted crystal in position. The saw must be rigidly mounted to keep vibration to a minimum. An efficient way is to mount the saw on a concrete block as shown in Fig. 2 and clamp the bed tightly by means of metal claws directly to the block.

The cutting angle is accurately controlled by x-ray measurements. A test piece is cut and a vertical arrow pointing up is marked on the outer surface to preserve the sense of direction. The test piece is measured by x-rays and the indicated horizontal and vertical angular adjustments made on the saw table. The procedure for correlating the readings is described in detail in an accompanying paper.[3] The saw should be regularly checked to make certain the angles are being maintained. Usual practice is to check every third or fourth wafer and correct the saw if necessary. With care, the X-block method and well-maintained saws, the saw maintains the angles within tolerance in wafering an entire crystal (10–20 cuts) once the saw has been corrected as indicated by the test cut.

When the crystals are irregularly shaped so that one or both of the bottom ends are not cemented to the plate, the wafering is started from one end and continued to the middle part cemented to the glass plate. The crystal is then translated to the opposite end and wafering continued from there. In this way butt ends of crystals do not fall off the mounting.

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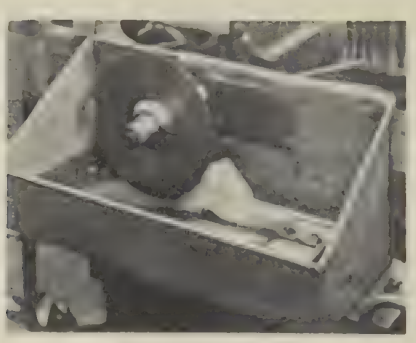
[3] Parrish, William, and Gordon, Samuel G., Precise control of quartz cutting with x-rays: *Am. Mineral.*, this issue.

Converted Milling Machines and Surface Grinders. Many types of expensive milling machines (Fig. 4) and surface grinders (Fig. 5) have been converted for sawing quartz but these have not had the success of the Felker-type machines. They all require expensive reconversion and generally are not designed for 24-hour operation. Most of them require cross-cutting which is not as good as down-cutting and allow little or no adjustment of the cutting angles. Off-angle wafers must therefore be



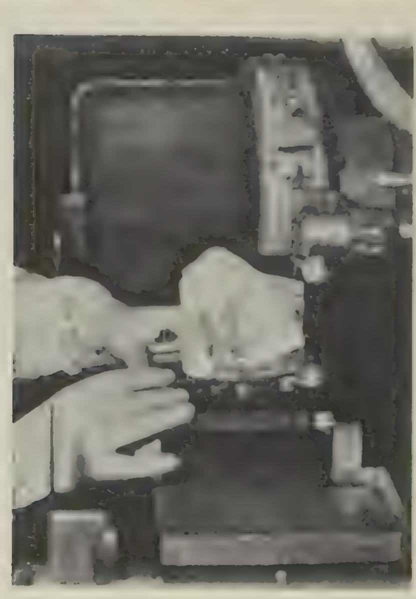


Fig. 4. Milling machine converted for quartz cutting.
Fig. 5. Surface grinder converted for quartz cutting. Magnetic chuck holds jig on which quartz is mounted.

corrected on a special lap of the type shown in Fig. 6. Machine-operated feeds are required to obtain smooth surfaces.

In the early days of the crystal industry, milling machines were used for "gang" cutting. Several blades spaced at equal distances were mounted between flanges on the same spindle. The blades were either diamond blades or metal discs rotating in a trough of wet silicon carbide ("muck" saws). The method has not proven practical for modern oscillator-plates with rigid angular tolerances because of the excessive vibration, difficulty in keeping the blades true, excessive waste of quartz, etc.

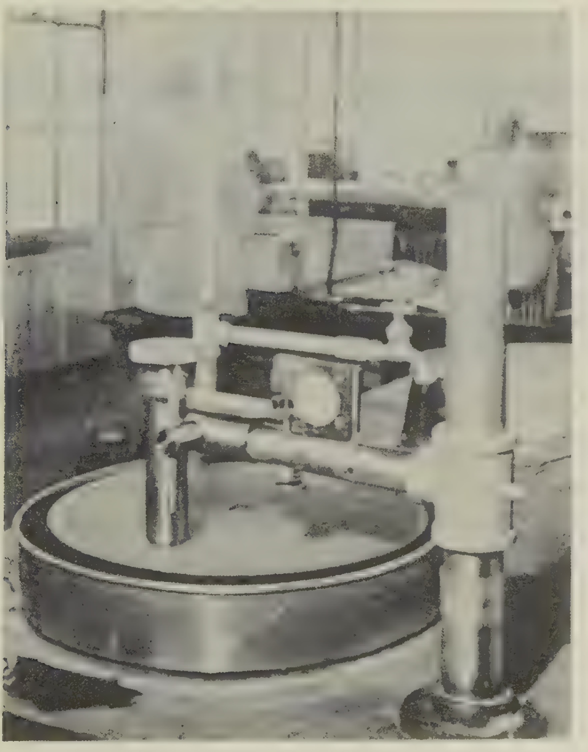
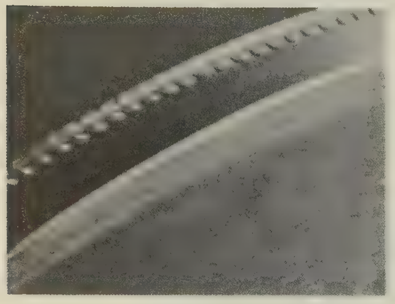


Fig. 6. Angle-correction lap. Off angle blanks are cemented to a plate which is attached to the arm. The plate is tilted with respect to the lap by the amount indicated by x-ray measurement and lapped true.

Diamond Blades

The "muck" type of saw is totally inadequate for the precision type of cutting required in the quartz crystal industry. Used in "gang" form in the early days for making X-cuts, they have been replaced by fine diamond blades. The two types most widely used are shown in Fig. 7. The outer rim of the blade first developed is notched, the diamonds rolled into the openings which are then pressed together to hold the diamonds by friction contact with the metal.[4] A relatively recent development is the metal bonded diamond blade in which diamond and a bronze powder are mixed, pressed and sintered by powder metallurgy methods onto the rim of a steel disc.[5] These blades are available in various diamond concentrations and grit sizes and the proper blade should be selected for the particular cutting operation. The life and quality of the cuts of both types of blades are dependent upon the quality of the sawing machine (amount of vibration, stability of spindle, etc.), type and amount of coolant, the crystal being cut (type of entering surface, stability of mounting, size of cut, etc.) and the cutting speeed (r.p.m. of blade, rate of down-feed).



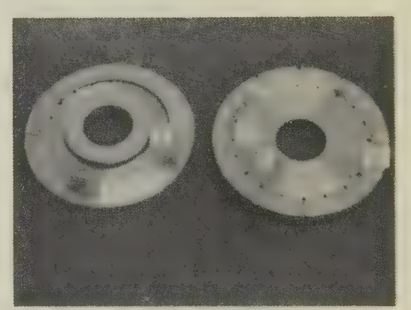


FIG. 7. Two widely used types of diamond blades. Front, Norton metal bonded blade; rear, Felker Di-Met notched blade.

FIG. 8. Flanges which are also used to apply coolant. See text for description.

There is a real need for thinner diamond blades. Weight losses up to 70–80% are caused in the wafering operations because the blade is generally thicker by a considerable factor than the wafer. Wafers must also be cut three to four times thicker than final thickness to allow enough quartz to be lapped off to remove tears and scratches caused by the blade.

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[4] This type of blade (Di-Met Rimlock) is manufactured by Felker Manufacturing Co., Torrance, Calif., and is available in steel (for fast cutting) and copper (slower cutting but longer life). Sizes: 3″ to 24″ diameter. Several crystal manufacturers have made their own notched blades but this practice is not common today.

[5] This type of blade is manufactured by Norton Co., Worcester, Mass. Sizes: 3″ to 14″ diameter.

Coolants. A copious flow of coolant on both sides of the blade is required. Mixtures of one-third to one-half deodorized kerosene and two-thirds to one-half light oil (such as transformer oil) are better than the water-soluble oil mixtures for the metal bonded blades.[6] One part of mineral (*not* vegetable) base water soluble oil to four parts of water (proportion is critical) is recommended by the manufacturers for the DiMet blade. The coolant is raised to the saw by means of a small circulating pump set in a reservoir with a series of baffles for settling out the quartz particles.

Flanges. The best results are obtained by using large flanges which should be at least one-third the diameter of the blade and preferably larger. The flanges may also be utilized in an efficient method of applying the coolant. A circular channel (A) with outer side sloping outward is cut into the flange, Fig. 8. The coolant is fed from tubing connected to the coolant tank directly into this channel. Holes (B) admit the coolant into reservoir (C) from which it is forced centrifugally into slots (D) which are flush with the blade. When the blade rotates, the coolant stays against the blade, does not splash and is directed into the cut.

Operating Conditions. The speed recommended for both types of blades is 4000 ± 500 s.f.m. With good sawing equipment and procedures, even higher speeds of 6000 to 7000 s.f.m. are used. The pressure is measured with the blade running and retardant open and should be small, not exceeding 5 to 7 lbs. The average rate of cutting with an 8″ blade under these conditions varies from 3 to 5 sq. in. per minute. A better finish is obtained with lower speeds and pressures but the blade is less efficient with present types of saws. In the metal bonded blades, the coarser the diamond grit, the faster the cut and the longer the blade life, but they produce deeper scratches. The following data were obtained for a 12″ Norton blade with 5″ diameter flanges, 1825 r.p.m., in cutting wafers of 5 sq. in. area:

Time	*Pressure*
92 sec.	9 lbs.
123	6
185	4

The truer the blade runs the better the wafer finish. The maximum allowable run-out on the o.d. of the blade is 0.002″ with maximum side run-out 0.005″. The edge of the blade must be kept symmetrical to minimize drift. Since it wears faster on the side towards the block, this may be accomplished by cutting one block starting from the left, the next block from the right and so forth. The Norton blades should be dressed lightly about every four hours, wet or dry, on the high corner with an abrasive stick but the side of the blade should never be dressed.

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[6] Although this mixture has a high flash-point, CO_2 fire extinguishers should be immediately available.

Drift of the blade is reduced to a minimum if the cut is started very slowly until the blade has cut about 1/32″ into the quartz. This is readily accomplished on the Felker #80 and #120 saws by holding back by hand the handle until the entering slit is made and then the speed is controlled by the hydraulic retardant. To prevent ridges, the down-pressure should not be varied once the cut has begun. The depth stop is adjusted so that the blade cuts slightly into the glass mounting plate to facilitate removing the wafer. The through feed is operated only after the blade has entered the glass plate to cut the front and back of the wafer.

Table 1[a]

Arc of Contact	Blade Size		Blade Specification		Flange Diameter	Minimum Wafer Thickness
	Diameter	Thickness	Grit Size	Concentration		
Up to 1½″	8″	0.045″	100	L25M	4 –5″	0.040″
Up to 2½″	8	0.050	80	L25M	3 –3½	0.045
2½″	8	0.055	60	L25M	2½	0.050
Up to 2½″	10	0.050	80	L25M	5	0.045
Up to 3″	10	0.055	80	L25M	3½	0.045
>3″	10	0.060	60	L25M	3 –3½	0.050
>3″	12	0.060	60	L25M	4 –5	0.050

[a] Data from Norton Co., Worcester, Mass.

Cutting Large Sections. In cutting X-planes and Z- or Y-sections and in rough trimming operations, a very thick blade with coarse diamond grit is required. This permits cutting into sloping surfaces, reduces drift and speeds the cutting and following etching operation. In this type of cutting, the thickness of the blade is of no importance because at this stage the yield is not yet a factor. The Norton D46-125M has been successfully used for this work.

Wafering from Thin Bars. Cutting blanks from Y- or complementary-bars is the simplest and most precise procedure. Since the arc of contact is very small and the entering surface is flat and perpendicular to the blade, a small, fine diamond grit size, thin blade with large flanges may be used. This is the ideal condition for minimizing blade drift and also allows cutting the blanks as thin as 0.035″, thus permitting a considerably greater yield for the same weight of quartz than is obtainable in wafering by the other procedures. When thin bars, the same size as blanks are cut, microscope slides or thin glass may be cemented to the surfaces

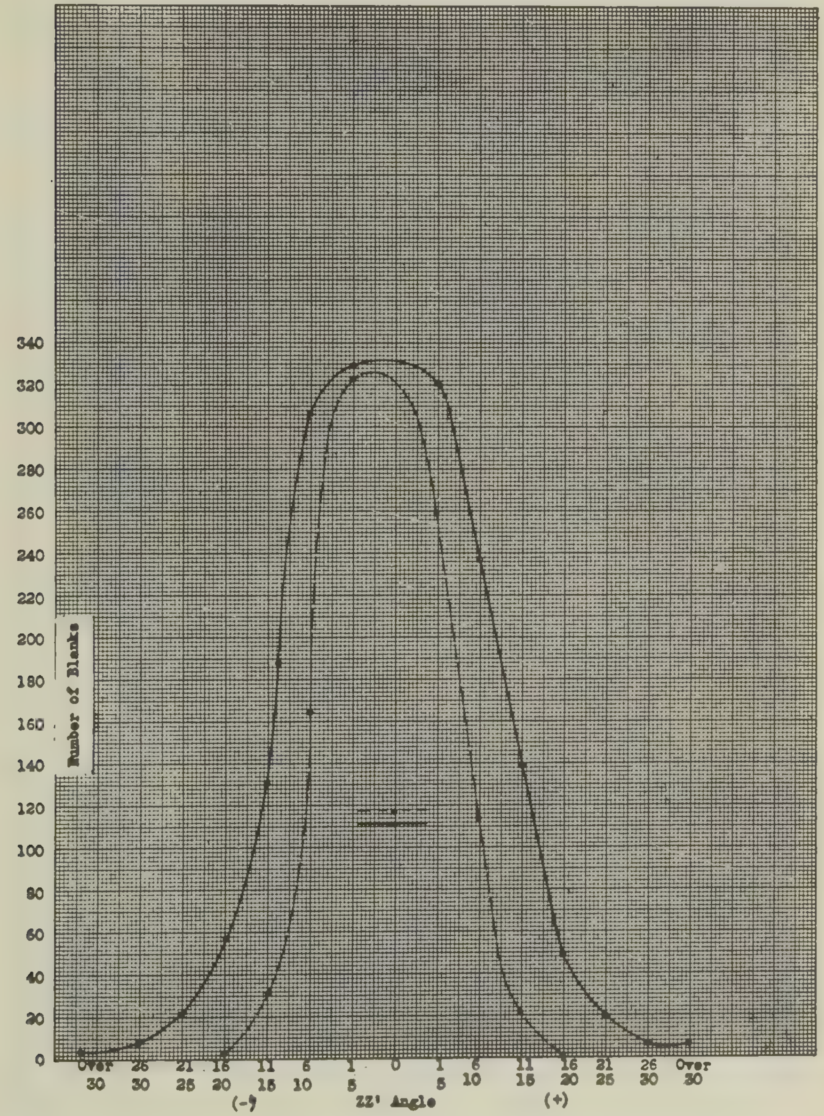

FIG. 9. Cutting accuracy. X-block method, Felker saws, Norton blades. Outer curve for ±15′ specified angular tolerance; inner dotted curve for ±10′ tolerance.

to prevent chipping of the blanks at the entering position of the blade or the sides. Two blades recommended by the Norton Co. for this procedure are 8″ dia., 0.045″ thick, 100S-L25M and 6″ dia., 0.035″ thick, 100S-L25M.

Wafering from X-Blocks. Wafering from X-blocks is an ideal set-up for cutting efficiency, precision and yield because the mount is firmly clamped to the saw table, thereby minimizing vibration. The entering surface is flat and perpendicular to the blade and the arc of contact is not excessive, so that thin blades and large flanges may be used (Table 1). About 5000 sq. in. would be a good life from a Norton blade used under these conditions.

The data given in Fig. 9 show the accuracy that is attainable with the X-block, wafering method. These results were obtained with Felker #80 and #120 saws using Norton metal bonded 8″ and 12″ diameter diamond blades. Cutting $\frac{1}{2}$″ sq. BT blanks with a specified angular tolerance of ±15′, 75% (1308) blanks were within ±10′ and 90% (1570) within ±15′. When the specification was reduced to ±10′, 94% (979) were within tolerance and 99% (1025) within ±15′.

It is also important to maintain a uniform thickness in cutting wafers. Too thick wafers are wasteful and when cut too thin cannot be used because the saw marks cannot be completely lapped off. Specifying 0.045″ ±0.005″, 87% (14,554) blanks were between 0.041″ and 0.051″ (Fig. 10).

Direct Wafering. Not only are the direct wafering single mount set-ups undesirable from the standpoint of orientation and yield as described in an accompanying paper, but they also are unsatisfactory because of the poorer blade performance obtained in cutting. The crystals are generally mounted on jigs which cause instability. Due to the irregularity in the shape of the entering surface, smaller flanges and hence thicker blades are required to minimize drift. Most manufacturers using such methods must angle-correct a considerable percentage of the blanks in order to meet the rigid angular specifications. Since the mount is usually at an angle to the saw bed, the saw stop cannot be set for one stopping point and the operator must be careful not to cut through the mount into the jig. In the X-block method, the depth stop is fixed to the same point for all the crystals, and hence one operator can look after several saws. 8″ and 10″ diameter blades, 0.045″ to 0.060″ thick, 60 or 80 grit size, L25M concentration are recommended by Norton Co. for this operation. The exact blade to choose is dependent upon the arc of contact; the smaller the arc, the thinner the blade and finer the grit size. The minimum wafer thickness should be 0.045″ to 0.050″, depending upon the blade used. If a considerable amount of angle correction is required it may be advisable to increase the minimum wafer thickness by 0.005″.

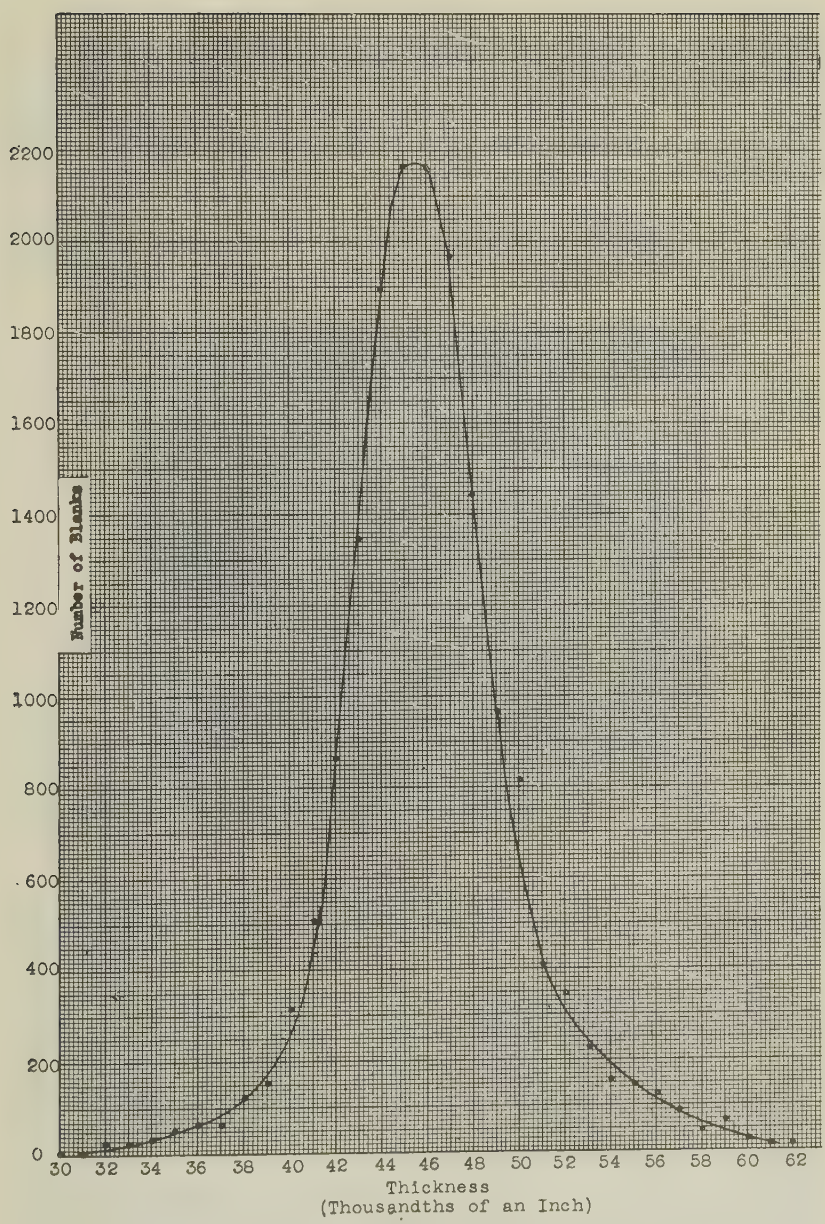

FIG. 10. Thickness distribution curve. Specified thickness 0.045 ± 0.005″.

TRIM SAWING (DICING)

The operation of cutting the blanks from the wafers is known as trim sawing or dicing. The wafers are etched, twinned and flawed areas marked out at the twinoscope, the usable portion of electrical twins determined, the wafer stauroscoped and the blanks laid out with a rubber stamp. The blanks are usually trimmed about 0.035″ to 0.045″ oversize to allow for squaring to final dimensions.

The usual method is to employ a trim saw of type shown in Fig. 11.[7] The blade position is fixed, the wafer held stationary by fingers on the saw table and the latter rocked into the blade. This arrangement has proven more successful than sliding the table or the wafer into the blade. The saw table (Fig. 12) has a pair of adjustable reference edges and a wide slit for the blade which is about $\frac{1}{4}''$ above the surface of the table. The back reference edge is perpendicular and the side reference edge parallel to the blade. The distance between the latter two is fixed to the desired blank width; a special arrangement permits adjustments in steps of 0.001″ up to 0.020″ without disturbing the reference edges by moving the slit laterally with respect to the blade.

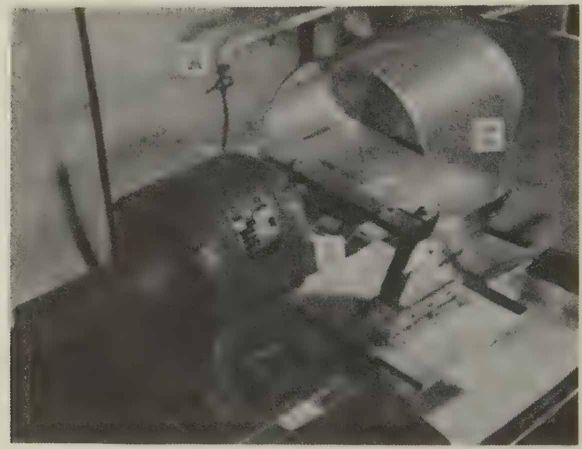

FIG. 11. Trim saw for dicing wafers. Table and wafer are rocked into blade. (A) Coolant system; (B) shallow felt filled reservoir for coolant; (C) coolant shield.

Wafers cut by the X-block method already have reference edges exactly perpendicular to X remaining from the X-planes. The reference edge permits maintaining the dicing angle parallel to the Z-axis. The smoothest edge is placed flush against the back reference edge and a cut made perpendicular to it along the edge of the first row of blanks. The wafer is then shifted laterally until the cut edge is flush with the side reference edge and strips of blanks are cut. The strips are cut into blanks in a similar fashion. The cutting is rapid. A 2″ long cut in a 0.045″ thick wafer takes 15 seconds. The average time for cutting a ½″ square blank is one minute.

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[7] Designed by T. W. M. Schaffers, N.A.P. and manufactured by Hammond Machinery Builders, Inc., Kalamazoo, Mich.

A 3″ dia. Norton metal bonded blade, 0.020″ thick, D180L50M is used at 3450 r.p.m. The coolant, a mixture of deodorized kerosene and transformer oil, is supplied by a small circulating pump to a shallow felt-filled trough around the bottom of the blade. Other blades used for trim sawing include 4″ and 6″ dia. 0.035″ thick. Resinoid bonded blades are also used but must be operated at much higher speeds.

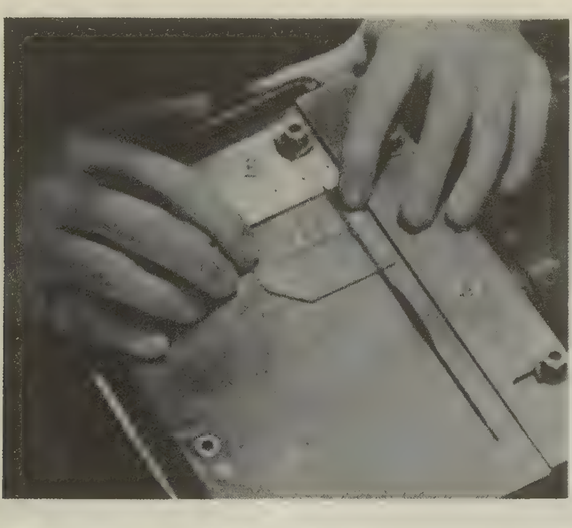


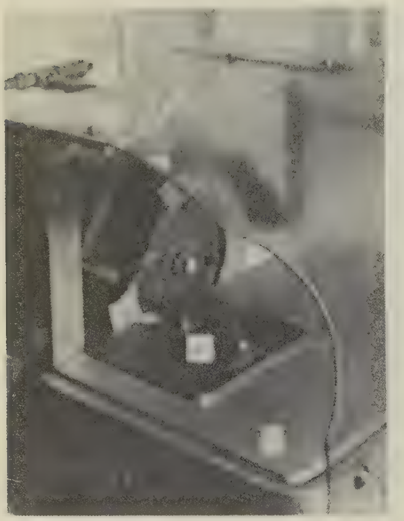
FIG. 12. Trim saw table. (A) Adjustable side reference edge set to desired blank width from blade; (B) rear reference edge perpendicular to side reference edge and blade. First cut is being made on wafer after which it will be translated to side reference edge to cut strips of blanks.

Another method of dicing is to cement the wafers, major face down, onto a large flat metal plate which has grooves a little wider than the diamond blade, spaced the trim-sawing distance apart. The plate is positioned on a saw table by means of pins on the under side of the plate and the wafers diced by cross-cutting. Although the method is faster than the one described above, the yield is much lower because there is no way of salvaging twinned and flawed areas.

SQUARING

The process of reducing trim-sawed blanks to approximately final edge dimensions is known as *squaring* or *edging*. It is one of the most difficult steps in the procedure. The blanks are often squared before the first lapping stage and the tolerance generally specified are ±0.001″; in predimensioning even closer tolerances are required and no completely satisfactory method has yet been developed for this purpose.

Crystals may be squared individually (when small quantities are required) or by loafing together (for production purposes). A machine for squaring crystals individually is shown in Fig. 13. A 6″ diameter metal bonded diamond cup wheel such as the Norton D150N100M mounted on an Excello grinder was used for this purpose. Resinoid bond wheels are also used and the new vitreous bond is promising. Various diamond grit sizes are available; the finer the grit used, the slower the cutting but the better the finish. Two stages of squaring are often used to obtain maximum speed and quality of finish. The wheels are run at a



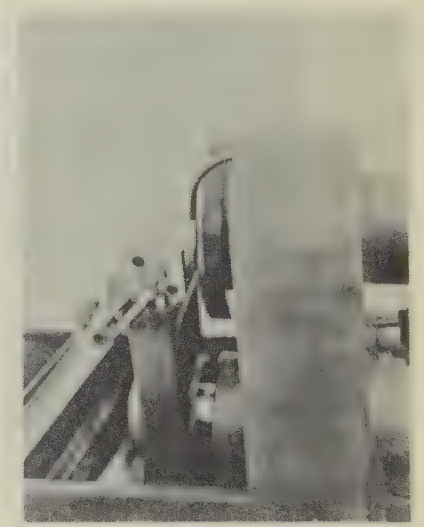
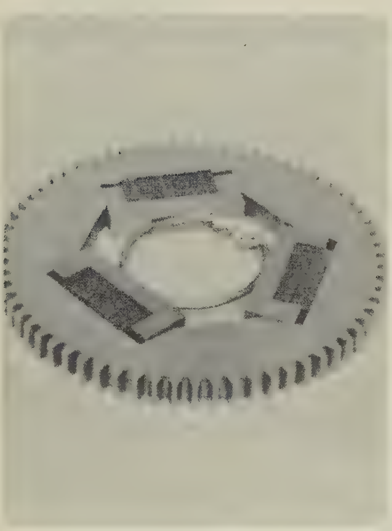


FIG. 13. Machine for squaring blanks individually. A 6″ dia. metal bonded cup wheel runs on an Excello precision grinder, 3450 r.p.m. The crystal is held in place in jig (A) whose position can be adjusted with respect to wheel. Large light weight table (B) runs on ball bearing races and is easily moved by hand. Micrometer screw working against stop permits setting to desired dimensions.

FIG. 14. New crystal edging machine manufactured by Volkel Bros. Machine Works, Los Angeles, Calif. Loaf of blanks carried by arm which oscillates over faces of Felker Di-Met resinoid bonded diamond abrasive wheel.

relatively high speed (3450 r.p.m.) and must be mounted on a good spindle to keep vibration to a minimum. The jig which holds the crystal on the table must be accurately set with respect to the wheel to obtain perfectly square blanks. The width of the blanks can be set by means of a micrometer screw working against a stop. Tables for machines of this type must be carefully designed and built to obtain good results.

Various methods of squaring by waxing crystals together in a loaf on a V-block are used. In some methods the V-block is held magnetically to the base of a surface grinder and one edge of the loaf lapped with a diamond wheel. After one side is finished the loaf is rotated 90° and the operation repeated. Some of the problems encountered are chipping of the blanks and difficulty of obtaining perfect right angles between the edges. The new Volkel crystal edging machine[8] was especially developed for this work. It is stated that one machine (Fig. 14) has a capacity of 2000–3000 crystals in eight hours. The crystals are mounted in a block on an arm which oscillates over a Di-Met resinoid bonded diamond cup wheel and automatically shuts itself off when the required size is attained.



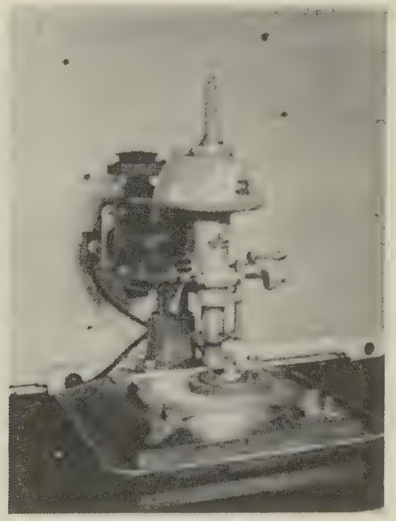


FIG. 15. Planetary lap gear for squaring crystals. Loaves of blanks are loaded in special gears and two surfaces lapped at once. Loaf is turned over for lapping other pair of sides. Method has extraordinary production capacity.

FIG. 16. Method of lapping-in diamond abrasive wheels. Backing plate screwed to wheel and lapped in a few minutes with drill press lap on glass plate with coarse silicon carbide. This "unloads" diamond points and sharpens cutting action.

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[8] Manufactured by Volkel Bros. Machine Works, Los Angeles, Calif.

An excellent production method devised by E. F. Sheeder, N.A.P., employs the planetary lap.[9] Crystals are held in U-shaped blocks without waxing and are tightened lightly from one end by a set screw. Three blocks are placed in a gear (Fig. 15) properly machined and three gears at a time are lapped. A spring ring holds the three U-blocks in position. The crystals are lapped with #320 or #600 silicon carbide and a dial micrometer on the upper lap plate indicates when the proper thickness has been reached. After one pair of sides is lapped the crystals are rotated 90° for lapping the other pair of sides. One planetary lap has a capacity of about 5000 blanks per eight hour shift.

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[9] Parrish, William, Machine lapping of quartz oscillator-plates: *Am. Mineral.*, this issue.

MACHINE LAPPING OF QUARTZ OSCILLATOR-PLATES

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ABSTRACT

A description is given of several types of machine lapping methods and equipment used in the manufacture of quartz oscillator-plates from the rough cut stage up to the final etching-to-frequency operations. The blanks are lapped from *ca.* 0.045″ thick to 0.012″ to 0.018″ depending upon the desired frequency and at the completion of the final stage the major surfaces must be parallel to within a few fringes of light, have a fine finish (usually with #303 optical flour), oscillate, and be close to final frequency (0.00004″ above final thickness).

A three-stage high-speed lapping-to-frequency procedure using planetary-type motion laps is described in detail. 30 to 55 crystals (depending upon their size) are carried in five revolving work-holders between two stationary lap plates so that both sides are lapped at the same time and rate. The weight of the top plate, speed, and abrasive size are progressively reduced in the three stages. A group of crystals is transferred from one stage to the next without intermediate grouping and one random transposition is made at *ca.* 0.0006″ from the final thickness to equalize thickness differences and contour on both sides. Total thickness variation is ±0.00002″. A calibrated sensitive radio receiver coupled directly to machine laps can be used to follow the progress of the crystals and to achieve precision control of the stopping point.

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* Mail address: Dobbs Ferry, N. Y.

Various other types of lapping equipment and procedures are described. The converted drill press lap is the most widely used in the crystal industry. The work-holder carrying the crystals is driven between a pair of stationary lap plates; the calibrating machine lap works on the same principle but is smaller. Optical-type laps have disadvantages for crystal work and are not commonly used. A special lap for single crystals and the milling technique in which as many as 1000 crystals at a time are tumbled, are described.

INTRODUCTION

This paper describes the machine lapping methods and equipment used in the manufacture of quartz oscillator-plates. The rough surface from the sawing operations must be removed and the blank properly lapped so that it will oscillate. The rough cut blanks are *ca.* 0.045″ thick and are lapped to 0.012″ to 0.018″ depending upon the desired final frequency.

Some of the several problems which must be simultaneously solved in the ideal lapping procedure are:

a. The lapped crystals must have the proper finish (usually with #303 optical flour) and contour (approximately 0.0001″ to 0.00005″ thicker in center than at four corners for 6 to 8 MC BT-cuts). The two major surfaces should be parallel to within a few fringes of light.

b. The crystal surfaces, edges and corners must be free from scratches and chips, and have the highest quality to prevent later difficulties in final finishing and use.

c. All crystals in each lap load must be closely alike in frequency at the completion of the final lapping stage. The approximate frequency spread should not exceed 10 to 15 KC for 6 MC BT-cuts which corresponds to 0.00004″. This eliminates the necessity of accumulating huge stocks of various frequencies, channel sorting, etc.

d. Recent experimental work has shown that 15 to 30 KC must be etched off 6 to 9 MC BT-cut crystals respectively, to prevent ageing.[1] Therefore each lap load of crystals must be brought to within 0.00004″ of final thickness without overshooting in order to reduce hand finishing to a minimum and obtain uniform crystals.

e. The entire lapping operation should be only a small fraction of the entire manufacturing cost. This means a large volume of high quality crystals, with a low rejection rate and hence the methods and equipment must be simple so that comparatively unskilled help can use them in a routine manner.

This study deals with the machine lapping of $\frac{3}{8}$″, $\frac{1}{2}$″ and $\frac{3}{4}$″ square BT-cut crystals in the 5 to 9 MC fundamental frequency range and $\frac{3}{4}$″ square AT-cut crystals, 2 to 5 MC. In these types of crystals the frequency is dependent upon the thickness

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[1] All lapped crystals age but the ageing is effectively stopped by etching off the disoriented surface layers after lapping. Frondel, Clifford, Final frequency adjustment of quartz oscillator-plates: *Am. Mineral.*, this issue.

$$\text{Frequency (KC)} = \frac{K}{\text{Thickness (inches)}};\ \frac{dF}{dT} = -\frac{K}{T^2}$$

and their relationship is shown graphically in Fig. 1. The frequency-thickness constant varies with the angle of cut, and for the angles used in this work the approximate values are: $K=100.7$ for BT-cuts, $ZZ'=49°30'$, $XX'=0°$; $K=66.5$ for AT-cuts, $ZZ'=+35°15'$, $XX'=0°$. In the higher frequencies, a minute decrease in thickness causes a large frequency increase so that the general practice is to make measurements directly in terms of approximate frequency (±2 to 4 KC) using a rapid

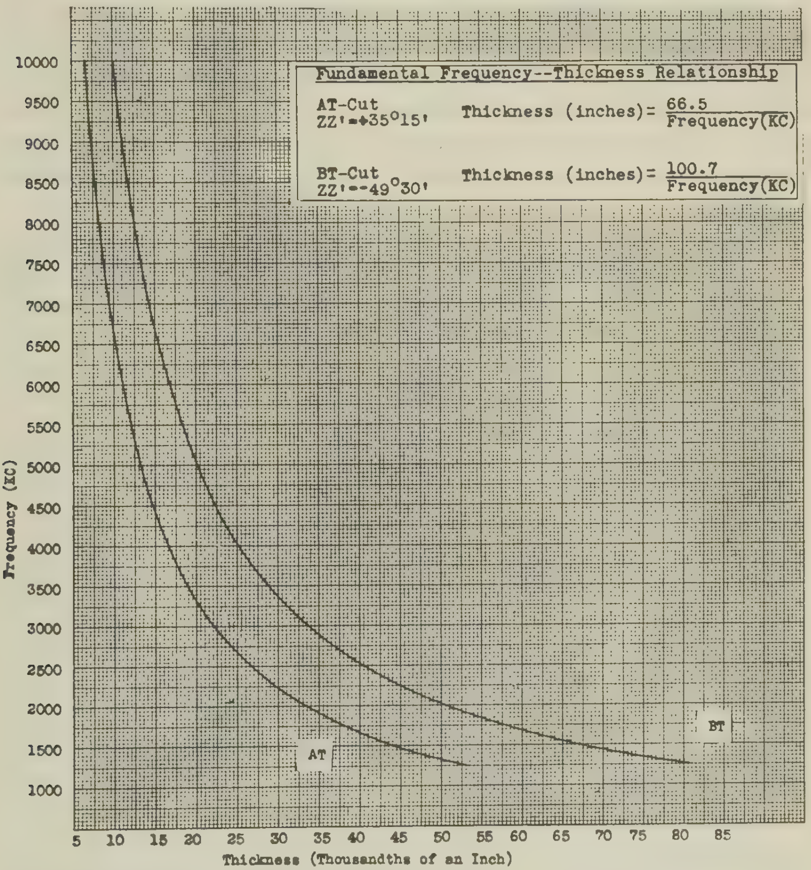

FIG. 1. Fundamental frequency-thickness relationship for AT- and BT-cuts.

direct reading device such as the channel sorter described later, rather than attempt precision thickness measurements.

It will be recognized that machine lapping of quartz crystals is a high precision job requiring a skillful procedure and good equipment. The sudden unprecedented demand for crystals after Pearl Harbor produced a variety of procedures and equipment hurriedly designed, with little opportunity to study even the barest principles involved, in order to get production started in the shortest possible time. Many of the fundamental problems have been solved and a few of the best practices will be briefly summarized. Some of the methods are applicable to the preparation of polished and thin-sections for mineralogical work, especially when used with the sawing procedures described in another paper in this Journal.[2]

Acknowledgments. The work on high speed planetary lapping to frequency was carried out under a development contract with the Signal Corps of the U. S. Army through the Signal Corps Ground Signal Agency. The work would not have been possible without their encouragement and support.

The writer is indebted to several engineers at N.A.P. for aid in this work: Mrs. S. Tinto[3] was in charge of the experimental work; the description of the radio receiver technique is based upon the development work of Mr. John D. Davies; Mr. T.W.M. Schaffers and Mr. E. F. Sheeder were responsible for the mechanical engineering; Mr. J. T. Derbyshire took the photographs; Mr. Charles E. Goldmann and several technical assistants carried out the detailed work. Mr. J. N. Bagwell, Jr. of Commercial Crystal Co., Lancaster, Pa. and Mr. P. R. Hoffman of Carlisle, Pa. were consulted on several problems. Bliley Electric Co., Erie, Pa. supplied Fig. 10 and information on the optical type lap. Atlas Sales Co., Chicago, Ill., supplied Fig. 12. Dr. Hal F. Fruth of Galvin Mfg. Corp. Chicago, Ill. reviewed the section on milling methods and contributed the data for Fig. 16.

LAPPING PROCEDURES

Only a few plants use a one-stage lapping process. A lapping stage refers to the grinding of crystals to a specific thickness with one size of abrasive. The three-stage process has proven the most successful although the four-stage and even the two-stage processes are in use.

The disadvantage of the single stage of lapping is that it requires a longer time than in the case of several stages. As the crystals acquire the finish of the abrasive in which they are being lapped, the lapping time increases enormously, particularly with the optical flours. It is practically impossible to lap rough cut blanks (0.045″) thinner than 0.020″ using only optical flour for the operation. In a two-stage process, the crystals are lapped with a coarse abrasive to a point where that surface can be removed with the final abrasive. A practical working process is the three-stage procedure with planetary laps tabulated in Table 1 using #320 and #600 silicon carbide in the first two stages and finishing with #303 optical flour (aluminum oxide).

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[2] Parrish, William, Methods and equipment for sawing quartz crystals: *Am. Mineral.*, this issue.

[3] Now at Research Division, Reeves Sound Laboratories, Brooklyn, N. Y.

TABLE 1. PLANETARY LAP INFORMATION

Lap Stage	1	2	3
Abrasive	#320 SiC	#600 SiC	#303 Al_2O_3
Outer ring speed (r.p.m.)	120–150	65–85	35–60[a]
Wt. top plate incl. collar (lbs.)	25– 28	20–22	15–18
Amount lapped off in one minute	0.0085″	0.0017″	0.0004″
Time for all operations, including loading, lapping, etc.	4 min.	6 min.	12 min.[b]
Stopping point	Final thick. +.011″	Final thick. +.003″	−10 to −30 KC from final freq.[c]
Stopping point control	Stopwatch, counter or radio	Counter or radio	Radio

[a] Variable speed control desirable in final stage.

[b] Includes time for transpositions.

[c] Transpose at frequency corresponding to 0.0006″ from final frequency; for BT-cuts, 6 to 8 MC.

The large frequency spread after the final stage and the use of laps carrying different quantities of crystals led to the grouping of blanks by thickness and/or frequency between each lapping stage. At the end of the procedure, those crystals which did not fall into immediately usable channels were either placed in stock or regrouped and relapped to a higher frequency. When the frequency spread occured again the process was repeated and in this way many crystals never reached the finishing department after having been relapped so many times that their frequency was too high for existing channels.[4] Grouping crystals did not permit a flexibility in scheduling, required much additional help and equipment, built up huge stocks and did not reduce the frequency spread.

Intermediate grouping is unnecessary if laps carrying the same number of crystals are used for all stages in the process. The only grouping required is to separate the rough cut blanks in 0.005″ groups so that they have approximately the same starting thickness. An ordinary hand micrometer is sufficiently accurate for the purpose. After the first stage of rough lapping, the crystals are kept as a group and transferred to the second stage, and then to the final stage. If some crystals are broken during the process, they are removed but none are added. At the end of the second stage the crystals are then automatically more closely grouped for thickness than is possible by any convenient production scheme now in use.

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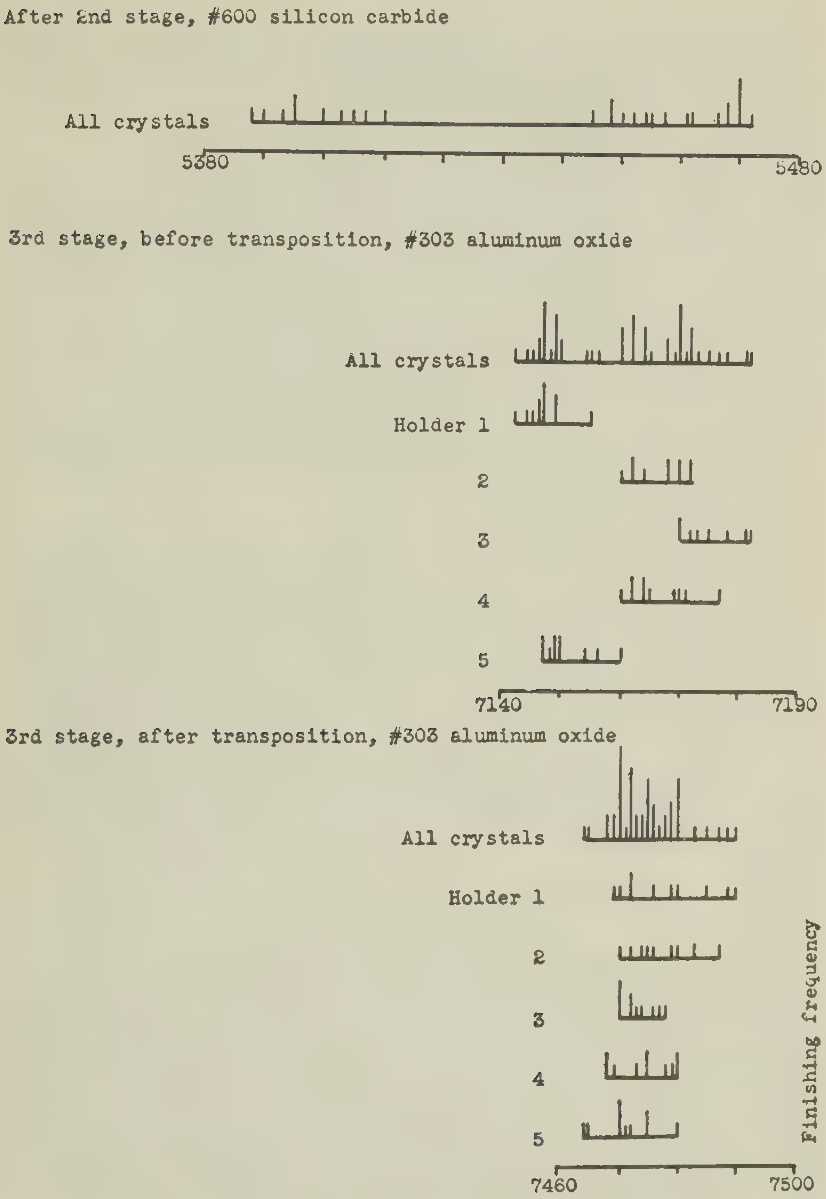
[4] This was referred to as the "lapping merry-go-round" in the trade.

Transpositions. One of the most difficult problems is to reduce the frequency spread to a point where the large majority of the crystals can be handled as one group in the finishing operations. The tolerable frequency spread will depend upon the frequency and finishing methods; 10 to 30 KC below final frequency has been the usual acceptable limit for 6 to 8 MC BT-cuts. This means that the crystals in a lap load must have the same nominal thickness to *ca.* ±0.00002″.

A detailed study of the problem was made with the planetary type of lap described below. This work involved lapping more than 10,000 crystals, 50,000 frequency measurements and numerous production data, so that it would be impossible to describe the numerous techniques tried. Using either three, four or five work-holders and either two or three stages of lapping, the frequency spread considerably exceeds the accepted limits. Spreads up to 200 KC were obtained on 8 MC BT-cuts but this was about the extreme, the average being 60 to 125 KC.

In order to determine the nature of the frequency spread the frequency of all crystals was measured after the second stage and the crystals from each work-holder were kept separate and measured at various times during the final stage. Numerous analyses of this type showed that the frequency spread in each work-holder was usually small, 10 to 20 KC, and the large spread was between the crystals in the different work-holders. This is illustrated in Fig. 2 where the smallest vertical line represents one crystal; the upper line in each stage gives the frequencies of all the crystals in the load and the smaller lines underneath show the frequencies of the crystals in the individual work-holders. Various patterns of frequency spreads were observed. Generally two work-holders had approximately the same frequency and the crystals in remaining work-holders were either higher or lower in frequency. No correlation between the pattern of frequency spread and lapping conditions could be made and it changed with time as the lap was in operation.

Since the spread in each work-holder is small, the spread for the run may be determined within close limits by measuring the frequency of one crystal from each work-holder. This can be done conveniently at the lap with a channel sorter of the type described below. If only a few work-


After 2nd stage, #600 silicon carbide

3rd stage, before transposition, #303 aluminum oxide

3rd stage, after transposition, #303 aluminum oxide

FIG. 2. Frequency spread within each work-holder is small but spread occurs between work-holders and is reduced by transposition of crystals. Smallest vertical line represents one crystal. $\frac{3}{8}''$ square BT-cut crystals.

holders are found to be at the desired end frequency, these crystals are removed from the lap and the remaining crystals of slightly lower frequency redistributed in the work-holders and the lap given a few additional turns to eliminate the spread. In this way frequency spreads were reduced from 53 to 18 KC, 70 to 30 KC, etc., at 8 MC.

It was determined empirically that if one random transposition was made in the final stage at a frequency corresponding to 0.0006″ from the final frequency that the spread was reduced to the required limits. The probable accuracy of this value is ±0.0002″ and should be redetermined for a new set of conditions. If the transposition is made too far from the final stopping point, the spread will again develop, and if made too close to the final frequency, the spread will not be sufficiently closed. It was found that several transpositions made during the procedure did not further reduce the spread but in transferring the crystals from one stage to the next a random transposition is automatically made. Figure 2 and Table 2 vividly show the effectiveness of the transposition technique in reducing the frequency spread.

Several types of transpositions were tried but a simple and useful one is to take all crystals from one work-holder keeping the upper surface face up, and stack them in the palm of the hand. This is repeated until all work-holders have been emptied and can be started from any work-holder and followed clockwise or counterclockwise. The entire group of crystals are then turned upside down so that the upper surface from the previous lapping will now be down. Starting with any work-holder, the top crystal from the stack is placed in the first work-holder, the second crystal in the second work-holder and so forth until the lap is filled. If some crystals are broken, the vacancies should be balanced on each side of the lap.

Transpositions are also useful in reducing the spread in the drill-press type of lap.

Abrasives. The most frequently used abrasives for machine lapping are #320 silicon carbide for the first stage, #600 silicon carbide for the second and #303 optical flour (aluminum oxide) or #800 silicon carbide for the final stage. #240 silicon carbide in water, laps almost two and a half times faster than #600.[5] Table 3 gives a summary of frequently used abrasive sizes. One-quart jars containing approximately 6 oz. of abrasive and filled with a lubricant such as Keystone Penetrating Oil #2[6] was used in the high speed planetary lapping method. The used abrasive is collected in

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[5] Bond, W. L., Processing quartz: *Bell Lab. Record,* **22,** 359–361 (1944).

[6] Keystone Lubricating Co., Philadelphia, Pa. have supplied the following information regarding this oil: gravity Baumé 35.2; flash point 225°F., fire point 280°F., Seybolt viscosity 56 sec. at 100°F., 33 sec. at 210°F.

TABLE 2. RESULTS WITH HIGH SPEED PLANETARY LAPPING PROCESS
ONE TRANSPOSITION, RADIO RECEIVER CONTROL

Blank Size	Cut	1st Stage #320 Silicon Carbide		2nd Stage #600 Silicon Carbide			3rd Stage—#303 Aluminum Oxide						Acceptable Range
							Before Transposition			After Transposition			
		Starting Thickness	Lapped To	Lapped Off	Spread	Lapped To	Lapped Off	Spread	Frequency	Lapped Off	Spread	Frequency	
3/8" sq.	BT	.044–.045"	.025"	.0053"	42 KC	5095–5137 KC	.0028"	10 KC	5974–5984[a] KC	.0005"	13 KC	6179–6192[b] KC	6170–6190 KC
"	"	.045–.047	.0263	.0070	d	5222[c]	.0024	27	5958–5985[a]	.0005	17	6179–6190	6170–6190
"	"	.040–.045	.0255	.0073	50	5515–5565	.0023	27	6348–6375	.0006	10	6580–6590	6580–6600
"	"	.042–.045	.024	.0064	d	5730[c]	.0026	21	6700–6721[a]	.0006	24	6978–6992	6980–7000
"	"	.039–.045	.0221	.0051	56	5878–5934	.0025	65	6905–6970	.0007	27	7238–7265	7240–7270
"	"	.040–.046	.0235	.0070	28	6102–6130	.0024	66	7100–7166	.0004	10	7330–7340	7330–7360
"	"	.046–.051	.0225	.0063	46	6214–6260	.0023	37	7225–7262	.0006	15	7595–7610	7580–7610
"	"	.045–.050	.0215	.0059	36	6464–6500	.0025	56	7654–7710	.0005	31	7959–7990	7958–7998
1/2" sq.	"	.045	.025	.0075	d	.0175"	.0024	d	d	.0005	23	6882–6905	6885–6905
"	"	.046	.026	.0085	d	.0175	.0027	d	d	.0006	16	7059–7075	7050–7080
"	"	.047	.024	.0066	d	.0174	.0026	d	d	.0006	20	7060–7080	7050–7080
.5 × .6"	AT	d	d	d	22	3335–3357 KC	.0033	39	4030–4069	.0006	16	4189–4205[b]	4185–4205
"	"	d	d	d	29	4058–4087	.0025	8	4808–4812	.0006	28	5090–5118[b]	5080–5120
.7" sq.	"	d	d	d	7	2892–2899	.0037	15	3438–3453	.0005	7	3539–3546[b]	3540–3550

[a] Measurements on one crystal from each work-holder.

[b] #303½ aluminum oxide.

[c] Only one crystal measured.

[d] No data recorded.

cans under the lap, filtered and the oil reused for rough lapping. The crystals must be cleaned with a solvent such as carbon tetrachloride to remove the oil. Three parts by volume of a water-soluble oil[7] to one part of Reprol[8] is reported to be successful and allows washing the crystals with warm soapy water. Good practice is to mix the abrasives by tumbling (see section on milling technique) and dispense it from an ordinary oil can. The crystals should be thoroughly washed between each lap stage to prevent contamination from the previous abrasive.

TABLE 3. ABRASIVE GRAIN SIZES[a]

Grit Size Designation	Average Grain Size in Microns		
	American Optical Co.	Carborundum Co.	Norton Co.
240			63
280		38	54
320		31.5	40
400		22.5	30
M302	22		
500		20	18
M302½	18		
600		17.5	12
M303	15		
700		14.5	
800		12.5	
M303½	11		
900		9.5	
M304	8		
M305	5		
F			44
2F			33
3F			23

[a] Data supplied by Companies noted. For smaller sizes see National Bureau of Standards "Simplified Practice Recommendation R118–40, Abrasive Grain Sizes," Aug. 15, 1940. See also *Optical Shop Eng. Bull.* No. 5, March 12, 1945, Off. of Chief of Ordnance, Fire Control Sub-Office, Frankford Arsenal, Philadelphia, Pa.

Thickness to be Lapped Off. In the final stage of lapping with #303 optical four, 0.003″ is lapped off the crystals to remove the #600 silicon carbide surface. In the intermediate stage 0.008″ is lapped off to remove the #320 surface. In the first stage the crystals are lapped from the original thickness to the final thickness plus 0.011″. Thus a load of crystals which is to be lapped to 6700 KC (approx. 0.015″) would be lapped to 0.026″ in #320, to 0.018″ in #600 and to final lap frequency in #303.

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[7] Sun Oil Co., *Sun Emulsifying;* Houghton Oil Co., *Permasol Oil.*

[8] Atlantic Refining Co., *Reprol.*

The amount to be lapped off the crystals in each stage was determined from the maximum grain size of the various abrasives. It was assumed that the maximum size of grain will produce a scratch of the same depth in the crystal. Since both sides are lapped, this value is multiplied by two and a safety factor of 100% added to each side so that the thicknesses were calculated on the basis of at least four times the maximum grain size of abrasive used. This procedure has been successful and assures that all lapped crystals have the same degree of optical flour finish regardless of frequency. Hence the crystals are properly prepared for the final etching-to-frequency operation. This is important for the lap finish markedly effects the etching time and where the finish is not controlled the etching rate is not predictable within the close limits usually required.

Quality of Lapped Crystals. Using the process described above, the crystals have good activity after the final lap stage so that hand edging at the finishing positions may be eliminated and the etching-to-frequency process is simplified. It is not uncommon to lap crystals to an activity of 0.4 to 0.7 where 0.3 ma. is passing in final test. Transpositions increase the quality of the crystals by producing crystals with a similar contour on both sides and decreasing wedging. Edge and corner chips and scratches (usually caused by contaminated abrasives) are the principal causes for rejection in the lapping process. The contour of the lapped crystals may be observed with a pair of high precision optical flats at least a few inches in diameter and a monochromatic light source such as a G. E. Sodium Lab-Arc. Quartz flats[9] are often used to prevent scratching the flats. The crystal is placed between the flats and the interference pattern observed at almost grazing incidence.

CONTROLLING STOPPING POINT OF LAP[10]

In all the lapping stages, it is important to be able to stop the lap at any desired crystal frequency or thickness. The need for precision control, particularly in the final stage, will be evident from the following discussion. If it is desired to lap a load of BT-cut crystals to 8000 KC, overlapping them by 0.0010″ will produce crystals approximately 700 KC higher than the desired frequency. In the final stage the stopping point tolerance is exceedingly small. 6 to 8 MC BT-cut crystals must be machine lapped to within 10 to 30 KC below the final finishing frequency which corresponds to a few one-hundred thousandths of an inch in thickness.

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[9] Acme Industrial Co., Chicago, Illinois.

[10] This section was prepared in collaboration with Mr. John D. Davies, N.A.P., Research Laboratory, who also did much of the development work.

The rate of lapping under a particular set of conditions may be determined by the use of a stop watch or an electric counter attached to the lap. Many carefully run tests have shown that these methods do not have the required precision in the final stage and that even experienced operators generally have to stop the lap at least a few times to check the thickness or frequency of a few crystals from the load in order to follow the progress of the operation. The lapping speed is dependent upon the grit size, composition and rate of feeding abrasive, crystal size and finish, weight of the top plate and speed of the machine.

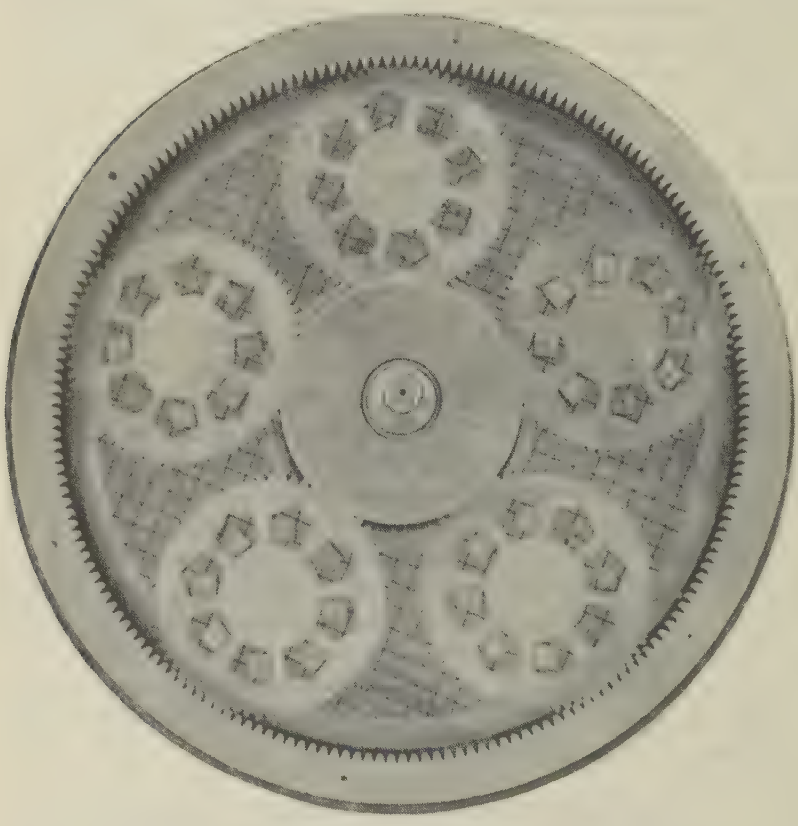

FIG. 3. Top view of planetary lap with upper lap plate removed showing crystals and work-holders.

A far more precise and simple method employing a sensitive calibrated radio receiver has been developed for determining the stopping point in all stages of lapping. With this technique the increase in frequency and the frequency spread of the crystals in the lap may be followed while the lap is in operation. The method is capable of high precision and eliminates guess work and difficulties in manual routine. It has been used for several years in a number of crystal plants but the writer is unaware of its origin.[11] The method as described here has been so refined and simplified that it may be placed in the hands of comparatively unskilled people with no background in radio who may be trained to use it successfully within a few hours.

In lapping with the planetary- or drill press-type of lap the crystals are carried by work-holders between the stationary upper and lower lap plates. Except for the thin abrasive and oil mixture, the lap plates rest directly on the crystals. During lapping the crystals are shock excited and emit a signal whose frequency is directly dependent upon the thickness-frequency constant of the type of cut (*AT* and *BT*, etc.) being lapped. This is an application of the familiar piezoelectric phenomenon of voltage produced by mechanical stress. The random voltages produced by the lapping action are sharply and strongly peaked at the thickness-frequency of the crystal (the crystal may be considered as a very high-Q tuned circuit.)[12]

A sensitive communications-type receiver is used to obtain the required delicacy of adjustments. In this work a Skyrider 32 (Fig. 4) was used.[13] The receiver-to-lap connections are shown in Fig. 5. The correct coupling is important because the average signal strength is of the order of a few microvolts and hence there is the problem of signal to noise ratio. Furthermore, since a sensitive receiver is used, the upper lap plate becomes an effective antenna and in the average industrial plant there is considerable electrical interference. One antenna post is connected to the upper lap plate and the other to the lower lap plate by means of the abrasive-guard shield which is more convenient than a direct connection. A common external ground is used for receiver and lap. Shielded cable is used for these connections to reduce the possibility of their acting as an antenna.

The upper and lower lap plates are separated by the crystals being lapped. Bakelite tubing is slipped over the two pins on the collar of the upper lap plate to prevent shorting to the lower lap plate. Bakelite workholders are used also for the purpose of preventing shorting. Zinc work-holders have been used when they are much thinner than the crystals for otherwise they short the two lap plates and the noise interferes with the signal coming from the crystals being lapped.

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[11] About a year after the experimental work was completed, a patent was found which described essentially the same method: Bailey, Richard S., Piezoelectric apparatus and method: *U. S. Patent Office* No. 2,340,843, Feb. 1, 1944.

[12] Van Dyke, K. S., The piezoelectric resonator and its equivalent network: *Proc. Inst. Radio Eng.* **16,** 742–764 (1928).

[13] Manufactured by The Hallicrafters Co., Chicago, Ill.

The receiver is calibrated at the frequency at which the lap is to be stopped by means of a calibrated oscillator loosely coupled to the former. A crystal oscillator with a standard crystal may be used but has the disadvantage of requiring a crystal for each desired frequency and also lacks flexibility. A simple and convenient method is to use a direct-reading calibrated variable oscillator, often called a *channel sorter* in the crystal industry (see Figs. 4 and 8). These have been built for the 1 to 5 and 5 to 9 MC ranges which covers the ordinary lapping frequencies. The variable oscillator has a large tuning dial marked to 5 or 10 KC and is calibrated with a standard crystal whose frequency is within a couple of hundred KC or less of the frequency at which the channel sorter is to be used. The standard crystal is plugged into the untuned Pierce oscillator circuit, the dial set to the frequency of the standard crystal and the variable oscillator calibration adjustment tuned to 0-beat. The standard crystal is removed, the dial set to the desired frequency and the signal is picked up by the receiver. The tuning dial of the receiver is set to the dial mark nearest the desired frequency (for a convenient reference point) and the receiver calibrated at this point by adjusting the bandspread dial to maximum deflection of the carrier level (S) meter. The receiver dial setting is then noted, as this is to be the final stopping point.

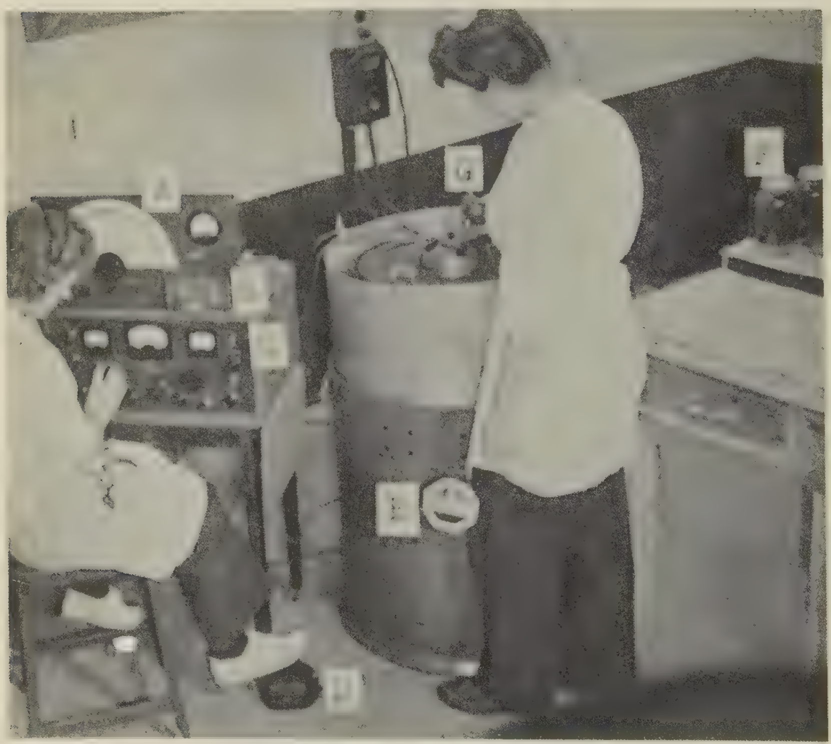

FIG. 4. Planetary lap equipment. (A) Channel sorter, (B) button electrode and anvil, (C) radio receiver, (D) foot switch for lap, (E) variable speed control for lap, (F) abrasive containers, (G) abrasive dispensers.

The electrical connections to the lap are put in place and the channel sorter dial turned far from the desired frequency so as not to interfere with the signal from the lap. With the R. F. gain turned up, the radio operator locates the starting frequency of the crystals when the lap operator turns the outer gear by hand at the beginning of the lapping operation. The radio operator starts the lap by means of a foot switch and follows the progress of the lap by turning the main tuning dial back and forth across the signal.

The best adjustments of receiver controls for lapping AT- and BT-crystals in the 4 to 9 MC region were found to be as follows: *Selectivity:* sharp IF; *BFO:* off; *Tone:* open; do not eliminate any of the high or low frequencies for signal contains both; *ANL* (automatic noise limiter): off, suppresses peaks when used; *Antenna trimmer:* adjust to final frequency or adjust while following signals across dial; *Carrier Level (S) Meter:* used only in adjusting receiver to standard signal; *AF Gain:* keep at full intensity; *RF Gain:* high when first locating signal then reduce to lowest volume for hearing; this retains sharp signal and keeps interference at a minimum.

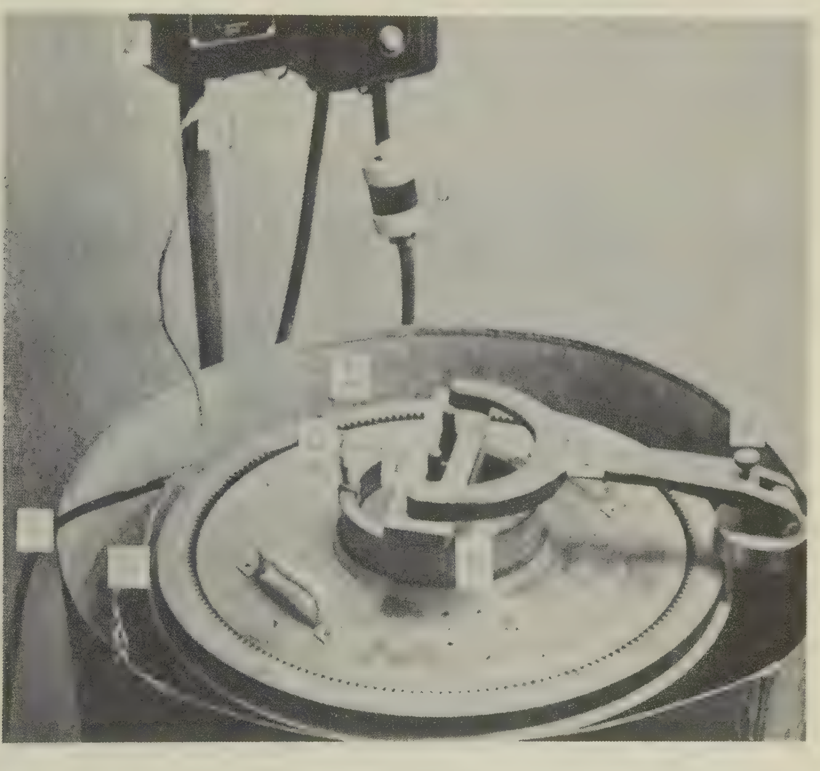

FIG. 5. Planetary lap. (A) Shielded cable, (B) antenna connection for lower lap plate, (C) antenna connection for upper lap plate, (D) ground connection, (E) bakelite bushing over collar pins, (F) main switch, (G) connection for foot switch, (H) special collar, (I) arm for holding upper lap plate in position, (J) screw stop for controlling vertical position of arm, (K) holes for feeding abrasive, (L) abrasive splash guard.

Head-phones, a loud speaker, or a recorder may be used to detect the signal but the first was found to be the most practical. In fact the stopping point may be automatically controlled.

The crystal signal from the lap may be recorded by connecting the 5000 ohm output terminals of the receiver through a rectifier (such as a 6H6 tube) to an Esterline-Angus 5 ma. DC graphic meter. A recording made in this manner is shown in the upper part of Fig. 9 for ½″ square BT-cut crystals in the final stage of planetary lapping with #303 optical flour. The receiver dial is set to the frequency at which the recording is to be made (B in Fig. 9). As the crystals are lapped thinner and their frequency approaches the dial frequency, the signal gradually increases in intensity, passes through a maximum and then falls off as the frequencies of the crystals pass the dial frequency. If the lap is stopped immediately and the crystals cleaned and channeled, their frequency distribution will be similar to the recorded curve as shown in the lower part of Fig. 9. The broadness of the left-side of the curve is caused by three crystals 25 to 30 KC lower in frequency than the other crystals, while the hump in the right-side of the curve is caused by all the crystals n one work-holder having a higher frequency than those in the remaining work-holders.

An additional advantage of the channel sorter used is that an anvil and button electrode in the Pierce oscillator provides a method of measuring frequencies directly to 3 or 4 KC, so that it may also be used for approximate frequency measurements after lapping. This further consolidates all operations into one position.

The radio receiver method may be applied to the lapping of other nonmetallic substances. For example, glass blocks could be lapped and their thickness followed by tuning in to a few quartz crystals of the same thickness placed in the same lap.

THE PLANETARY LAP[14]

This is the most successful lap that has been developed specifically for quartz crystals. When properly used it is capable of high precision and enormous production. By making one transposition in the final stage the frequency spread is less than 15 KC for 6 MC BT-cut crystals. This corresponds to 0.00004″ difference in thickness between the thickest and thinnest crystal in the load. Daily production from five or six planetary laps (one for #320 abrasive, one or two for #600 and three for #303 final stage) operating 20 hours per day is well over 6000 lapped blanks passing all specifications with a rejection rate for all causes less than 10%.

The name planetary lap has been adopted because the crystals undergo a planetary type of motion during lapping in this type of machine. It is also called the Hunt-Hoffman lap, after G. C. Hunt and P. R. Hoffman of Carlisle, Pa., who developed it.[15]

The crystals contained in either bakelite or zinc work-holders ride between the lap plates which are stationary. The periphery of the work-holders is toothed so that they mesh with the outer and inner ring gears between the lap plates. The ring gears, driven by a $\frac{3}{4}$ h.p. 3-phase motor in the under part of the housing, drive the work-holders and cause them to rotate and produce a broad sweeping lap motion. In addition, each crystal is free to rotate within the pentagonal hole of the work-holder and this produces the spinning motion.

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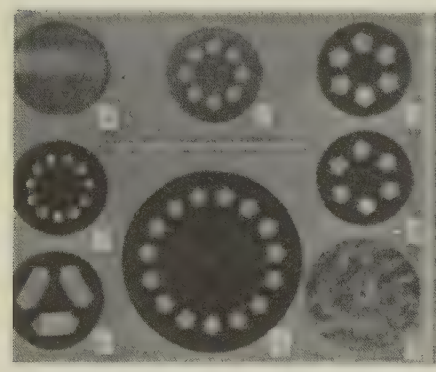
[14] Manufactured by P. R. Hoffman Co., Carlisle, Pa., and New Jersey Wire Stitching Co., Camden, N. J.

[15] Hunt, Grover C., Grinding machine: *U. S. Patent Office* No. 2,314,787, March 23, 1943. Hoffman, P. R., Grinding machine: *U. S. Patent Office* No. 2,308,512, June 19, 1943.

Work-Holders. The selection of proper work-holders is important in planetary lapping. Large frequency spreads, poor contour, low activity, chipping, etc., may result from the use of poor work-holders. Two types of material are widely used: linen-base bakelite and zinc. Although the latter has greater strength, it is a conductor and can be used with the radio method only when the crystals are considerably thicker than the work-holders for otherwise the two plates will be shorted. Bakelite is preferred also because it produces less chipping, less wear on the gears and less damage in a crack-up. Several types of work-holders are shown in Fig. 6.



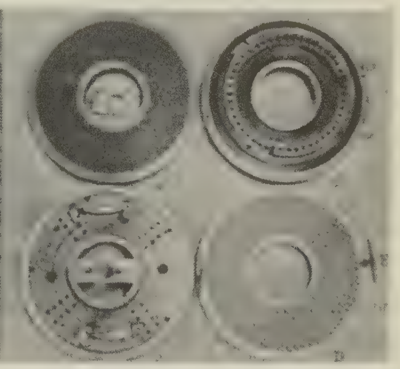


FIG. 6. Work-holders for lapping crystals. (A) Planetary lap, blank, (B) planetary lap, $\frac{1}{2}''$ crystal, straight side, (C) planetary lap, $\frac{3}{4}''$ crystal, straight side, (D) planetary lap, $\frac{3}{8}''$ crystal, straight side, (E) planetary lap, $\frac{3}{4}''$ crystal, curved side, (F) planetary lap, for lapping ultrasonic plates, (G) drill press lap, $\frac{3}{4}''$ crystal, curved side, (H) calibrating lap $\frac{1}{2}''$ crystal, slightly curved side.

FIG. 7. Planetary lap plates. (A) Lap surface of upper plate, (B) top view of upper plate, (C) bottom view of lower plate, (D) lap surface of lower plate, (E) removable handles for changing gear position of lower plate.

One of the principal difficulties in using bakelite work-holders is that they tend to become warped. Paper-base, double-and higher-ply, and single-ply linen base bakelite are used and they are listed in increasing order of their tendency to warp. Paper-base bakelite is so weak it can only be used with very light weight top plates such as in the small Bagwell planetary lap[16] or the Calibrating Machine Lap. Warped holders can sometimes be used by wetting them thoroughly with the abrasive mixture and placing the crystals in the middle of the hole rather than against the sides when loading the lap. When the top plate is put in place it will flatten the work-holder so that the crystals may not slip under the hole and cause a crack-up.

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[16] A small planetary lap employing small work-holders and a light weight top plate developed by J. N. Bagwell Jr., Commercial Crystal Co., Lancaster, Pa.

All five work-holders for the planetary lap should be grouped for thickness within 0.001″. Zinc work-holders should be lapped-in without crystals until most of the high spots disappear. Bakelite work-holders should not be lapped-in because it weakens their teeth. The work-holders should be at least 0.002″ thinner than the final lapped thickness of the crystal.

Pentagonal holes with sides slightly larger than the longest edge of the crystal allow the crystal to rotate during lapping and produce crystals with a proper contour for high activity. The corners may be cut back to reduce chipping the corners of the crystals. Curved-side pentagonal holes are also often used but the relative merits have not been rigorously determined.[17] Square holes in which the crystal fits snugly are undesirable because the crystals become wedge-shaped. The holes in which the crystals are contained must be so positioned that the crystals slightly over-ride the inner and outer periphery of the lap plate. This prevents excessive lapping of the edges and permits control of surface contour by varying the amount of over-ride.

Thurston has described a novel work-holder which may eliminate edge chipping.[18] The crystals fit snugly in a square hole cut in a small circular disc. The discs fit into a work-holder of the same thickness which has six circular holes just large enough to accommodate the discs and allow them to rotate during lapping. This paper should also be consulted for a description of a newly developed lapping machine for quartz crystals.

Lap Plates. Both upper and lower lap plates (Fig. 7) are normalized meehanite, 15 5/8″ in diameter with a 5½″ diameter center hole. The plates are used as a pair and should lie one on top of the other at all times to prevent warping and contamination. A pair of plates is used for each abrasive. Four series of extra radial holes countersunk at the top, are drilled in the upper lap plate to allow a more even feeding of the abrasive. Both plates have square serrations on the lapping surfaces which are ¼″ apart on the upper plate and ½″ apart on the lower plate. No published study of the effect of various types of serrations on lapping quartz has been made. Cast iron lap plates with serrations lap crystals almost twice as fast as smooth plates and as fast as smooth hard tool steel plates.[19] Glass lap plates are faster than smooth cast iron plates but the crystals and work-holders tend to stick to the lower surface of the upper plate when removing it from the machine. In addition to increasing the lapping speed, the serrations allow a more even distribution of abrasive so that the entire surface of the crystals receives an even lapping action and the edges are not lapped thinner than the center.

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[17] Hunt, Grover C., Work-holder for grinding machines: *U. S. Patent Office* No. 2,309,080, Jan. 19, 1943.

[18] Thurston, G. M., Flatness and parallelism in quartz plates: *Bell Lab. Record*, **22**, 435–439 (1944).

[19] Bond, W. L., *op. cit.*

Both plates have inner ($\frac{15}{32}$″) and outer rings ($\frac{11}{16}$″ wide) which should be at least 0.005″ and not more than approximately 0.010″ below the lapping surface of the plate. The rings permit the crystals to slightly overlap the inner and outer edges of the lapping plate surface so that the latter will remain flat out to the edges. As the plate wears, the rings must be pounded back into position with a soft hammer.

An upper lap plate weighing approximately 25 to 28 lbs. is used for coarse lapping with #320 abrasive. Progressively lighter weight plates should be used for the intermediate and final stages of lapping to permit the use of thinner bakelite work-holders and reduce excessive breakage of crystals and damage to the plates in crack-ups. Upper lap plates weighing 20 to 22 lbs. have been used for the intermediate lapping stage with #600 abrasive. In the final stage of lapping with #303 optical flour, a greatly reduced plate weighing only 15 to 18 lbs. is required if thin linen-base bakelite work-holders are to be used. These values include the weight of a 3 lb. special collar which has been added (see description below). The exact weight does not appear to be critical.

The flatness of the plates can be quickly determined by removing the plates from the lap and lapping by hand one against the other with the same abrasive as is used with these plates for a few minutes. Low areas then become apparent when the plates are separated and the lap surfaces inspected. A flat piece of accurately ground stock laid across the diameter of the plate on four small pieces of paper placed on the inner and outer periphery of the lap surface on both sides of the center hole may be used to measure the unevenness of the plate. If the plate is perfectly flat, the pieces of paper cannot be pulled from under the stock when the latter is held firmly in place. If one or more can be pulled out, the departure from flatness at those points may be determined by using two or more pieces of paper until they cannot be pulled out and then measuring the total thickness with a micrometer.

Before being placed in use, the lap plates are ground flat on a large surface grinder and then lapped-in with the same abrasive as will be used on those plates. For this purpose three gears the same size as the work-holders but about $\frac{3}{4}$″ thick with the center milled out leaving a $\frac{1}{2}$″ ring gear are used. The gears are put in place and the lap run until the plates are flat.

If crystals or work-holders break during lapping, the plates and gears must be thoroughly cleaned with a hard brush and the serrations with an awl. Cloths and towels should not be used on the plates for lint and stringers tend to collect in the serrations.

A bothersome feature of the planetary lap is that the gears which drive the work-holders, particularly the inner gear, become slotted by the work-holders. The position of the lap plate with respect to the gear must therefore be changed after several runs to prevent excessive slotting; the change is required more frequently with coarse than with fine abrasives. A pair of tapped holes in the outer ring of the lower lap plate which will admit a tool to lift the plate from the carriage will expedite the change. The plate is then lifted and turned to the next position, the latter being fixed by a step-like arrangement on the under side of the plate (Fig. 7). In this way the wear on the gear is equalized, and after it becomes worn the gear is turned upside down and used in the same way for only one side of the teeth become worn.

A preferred method of holding the top plate in position is shown in Fig. 5; it permits the ppper plate to float freely on the lower plate. A collar around the inner hole, with a cross-piece with slot and two vertical pins has been added to the lap plate; this adds approximately 3 lbs. to the weight of the plate. A slotted arm fits across the pins and is prevented from resting directly on the upper lap plate by an adjustable stop.

Operation of Lap. It must be emphasized that constant attention to minute details and a set routine such as checking ring depth, setting plates to new gear position, careful washing and inspection of crystals between stages, etc., are required to obtain good results. The work-holders should be checked and evenly spaced in the lap. The crystals are placed against the side of the pentagon, or in the middle if the work-holder is warped. Abrasive is squirted between the work-holders, the top plate and center arm put in position and more abrasive poured through the feed holes in the top plate. The outer gear is rotated counterclockwise by hand several times with the operator listening for any sign of a crack-up. The radio operator runs the lap by means of a foot switch and watches the progress of the lap by turning the main tuning dial of the receiver back and forth across the signal while the lap operator feeds abrasive at regular intervals. At the end of the run, the spread may be determined by measuring one crystal from each work-holder with the channel sorter.

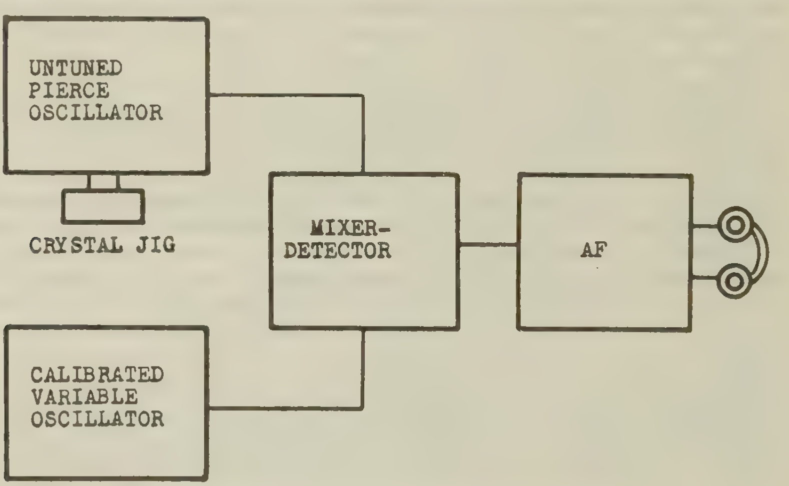


FIG. 8. Simplified block diagram of channel sorter.

MISCELLANEOUS LAPPING EQUIPMENT

There has been no published extensive study of the lapping procedures in current use in the crystal industry. The following descriptions are based upon observations of several dozen plants mainly in the East. Space and time limitations allow a brief description of only a few of these.

Optical-Type Lap. The crystals are cemented to a flat plate and one side is lapped (Fig. 10). The crystals are then removed, the lapped side cemented to a plate and the second side is lapped. This type of lap has certain disadvantages for quartz work and is used by only a few plants in the crystal industry. It may prove to be useful in the preparation of very thin plates, which have been previously lapped by the usual procedures to approximately 0.010″.

The "machine employs a 15½″ diameter cast iron lap rotating at a speed of 17.2 r.p.m. against which is driven at a speed approximately 99 r.p.m. the crystal mounting plate. For the average crystal work this mounting plate is 9½″ diameter and approximately 1⅛″ thick and is also made of cast iron and is lapped flat on both sides and parallel to approximately .000010″ over the entire usable surface.

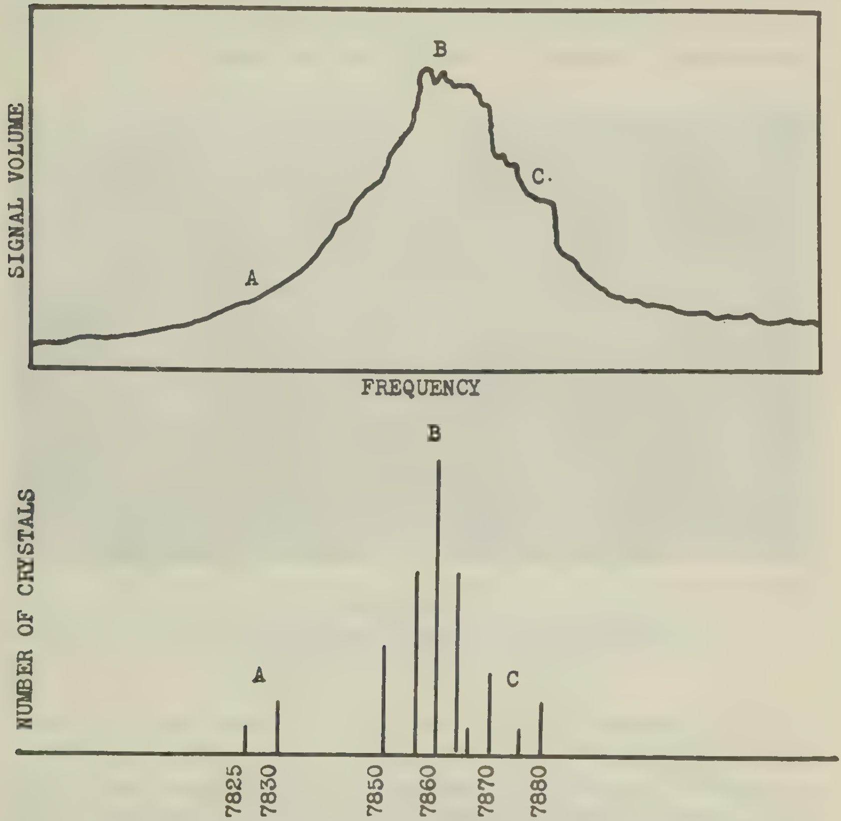
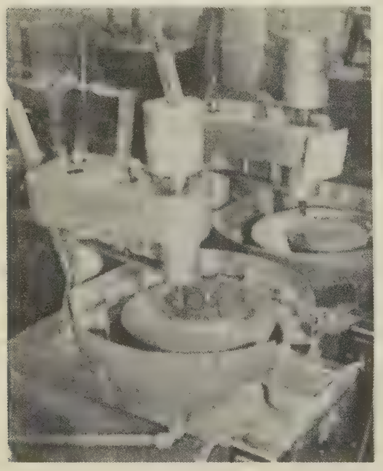


FIG. 9. Graphic recording (above) of crystal signals from final stage of planetary lapping with #303 optical flour. Receiver dial set at frequency B. Lap stopped immediately after recording and individual crystals measured with frequencies shown below. Smallest vertical line represents one crystal.

"The crystals are mounted on this plate with paraffin, taking care on the last side lapping especially to have an absolute minimum quantity of paraffin on the surface and carefully controlled heating. In this way we can control the mounting error so that only a very small percentage of the crystals on the plate will be out of parallel by more than .000020″ from corner to corner. The mounting plate is driven by means of a crank pin engaging a hardened steel center insert in the mounting plate. The throw of the crank as well as its center of rotation in relation to the lap are varied to maintain a flat working surface on the lap as well as to control the flatness across the mounting plate and the contour of each individual crystal. The work on the mounting plate is measured by means of a specially designed ring gauge employing a Pratt-Whitney Electrolimit Comparator Head."[20]



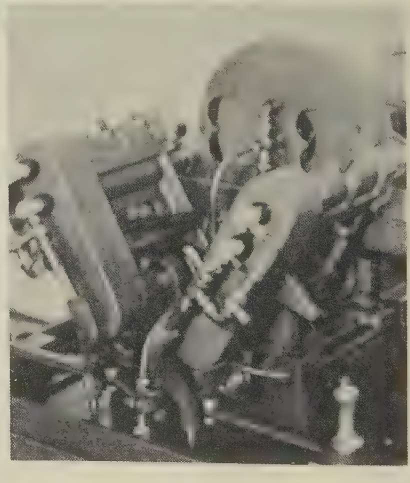


FIG. 10. Optical-type lap. (Courtesy of Bliley Electric Co., Erie, Pa.)
FIG. 11. The Q-lap. Crystal is held by vacuum chuck on rocker arm which oscillates across face of diamond grinding wheel.

Q-Lap.[21,22] This unique lap employs two rocker arms, each holding two crystals, which slowly oscillate over the face of a diamond abrasive wheel (Fig. 11). One side of the crystal is held by a suction device on the face of the rocker arm while the other side is lapped. The angular position of the crystal is slightly displaced with each successive cycle of the rocker arm and the rotation of the crystal holder at 16 r.p.m. produces a uniform grinding of the crystal surface. A stop nut which can be set in steps of 0.00025″ prevents grinding beyond the set thickness, after which the handle is retracted and the rocker arm locked in the idling position. Best results were obtained with a resinoid bonded diamond wheel, $8\frac{1}{2}''$ diameter, 100-grit size, #50 concentration, running at 1750 r.p.m. and a mixture of 20–30% turpentine and 80–70% kerosene by volume for coolant.

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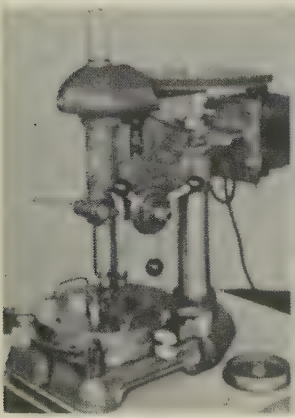
[20] Personal Communication, C. C. Collman, Bliley Electric Co., Erie, Pa., Feb. 12, 1945.

[21] Schaffers, T. W. M., The Q lap: *Communications*, **24**, 40–42, 80 (1944).

[22] Manufactured by Hammond Machinery Builders, Inc., Kalamazoo, Mich.

The Q-lap has been employed by only one crystal manufacturer. Its principal uses have been in grinding thick wedge-shaped blanks to parallelism with a reference surface, reducing extra thick cuts to a nominal thickness for lapping and in correcting off-angle blanks. In making test-cuts in wafering, the first wafer is usually wedge-shaped with the outer surface off angle and the inner surface on angle. The correct surface is attached to the crystal holder and the off-angle surface lapped to parallelism with the correct surface. Where both surfaces are off-angle, an adjustable head whose surface may be varied with respect to that of the diamond wheel is slipped on the rocker arm for making the correction indicated by x-ray measurement.



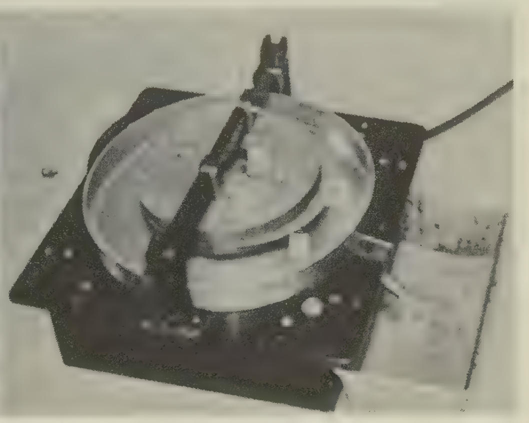


FIG. 12. Drill press lap. The most widely used type of lap in the crystal industry. (A) Lap plates, (B) work-holder, (C) offset arm, (D) rods for holding upper lap plate, (E) disc for lapping-in plates. (Courtesy of Atlas Sales Co., Chicago, Ill.)

FIG. 13. Calibrating machine lap. (A) Lap plates, (B) arm for holding upper lap plate, (C) offset arm, (D) abrasive guard shield.

Drill Press Lap.[23] A conversion of a standard bench type drill press (Fig. 12) has become the most widely used lap (about 2000) in the crystal industry. The adaptation was made several years ago by Western Electric Co.

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[23] Manufactured by Atlas Sales Company, Chicago, Ill., and William A. Hardy Co., Inc., Fitchburg, Mass.

The standard movable table and drill chuck are eliminated and the steel column holding the motor mount has been shortened. The lap plates are stationary and a single work-holder carrying from 12 to 20 crystals (depending on the size of the crystals) is driven between the plates by an off-set arm from the main spindle. The length of the offset arm is adjusted so that the crystals in the work-holder slightly overlap alternately the inner and outer edges of the lap plates. The bushing holding the work-holder (not visible in Fig. 12) fits loosely so that the latter rotates slowly about its own axis during lapping. The motion of the crystals is less complicated than in planetary lapping. Speeds varying from 30 to 100 r.p.m. have been used but 55 r.p.m. has been the average. The lap plates are serrated, 10¾″ diameter with 4¾″ central hole. The upper lap plate, weighing approximately 16 lbs., is held in place by four rods in U-shaped holders which alllow it a slight amount or no lateral play. Abrasive is fed through holes in the upper plate. An adapter disc fitting into the central hole of the lap plate is used to lap in the plates. The top and bottom plates may be interchanged a few times when lapping-in to obtain flatter surfaces.

Several complications have been introduced in attempting to improve the quality and speed of the drill press lap but they have not been generally adapted. In one of these, the upper and lower plates were made to revolve counter to each other at different speeds. This caused more to be lapped off one side of the crystal than the other and often saw marks remained on one side of the crystal. The radio receiver method of controlling the stopping point has been applied successfully to this lap when an insulated work-holder such as linen-base bakelite was employed and the four rods holding the upper lap plate are insulated so that the two lap plates are not shorted. Transpositions decrease the frequency spread and improved quality.

Several manufacturers are of the opinion that the drill press lap is not capable of the enormous production and high quality crystals obtained from the planetary lap.

Calibrating Machine Lap.[24] The principal of this lap is the same as that employed in the drill press lap described above but it is smaller (Fig. 13). The lap plates are serrated, 6½″ diameter with 2″ central hole. One work-holder is driven at 95 r.p.m. by an offset arm attached to the motor drive. By changing the length of the arm, the overlap of the crystals on the inner and outer edges of the lap plates may be varied. The work-holder carries 6 to 10 crystals depending on their size. Since the upper lap plate weighs only 3½ to 5½ lbs., thinner work-holders may be used with this type of lap than with the planetary or drill press so that it may be useful for thin crystals. A disc fitting into the central hole of the upper lap plate is attached to the eccentric arm for lapping the plates together when the work-holder is removed.

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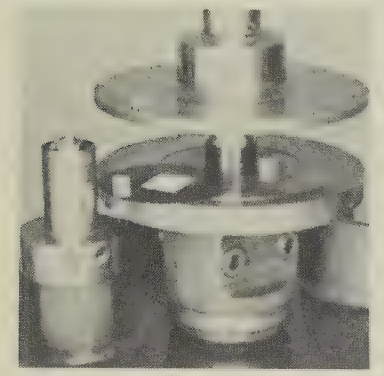
[24] Manufactured by Empire Electronics Corp., New York City.

Seco Precision Crystal Finisher Type F.T.[25] This small slow speed lap was developed for finishing single crystals (Fig. 14). The lap plates have a smooth disc-like plateau which is slightly narrower than the width of the crystal and is slightly above and extends around the entire lap plate.



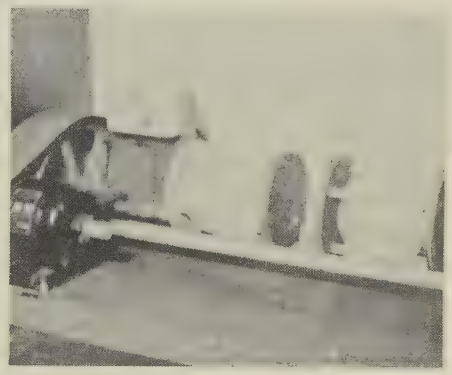


FIG. 14. Seco precision crystal finisher type, F.T. lap for single crystals. (A) Upper lap plate raised from lapping position, (B) lower lap plate, (C) work-holder with crystal, (D) post.

FIG. 15. Milling equipment for tumbling several jars.

The crystal is contained in a circular work-holder with a square hole just large enough to hold the crystal. The lap plates and post rotate clockwise and the work-holder counterclockwise. A timer is generally used to control the lapping time but the lap may be adapted for use with the radio receiver method. It is used after the standard lapping procedure (with planetary or drill press laps) to bring crystals to the same starting frequency for etching and to eliminate hand finishing at the finishing positions. It is also useful when only a few crystals of special frequencies are required.

MILLING TECHNIQUE

An ingenious approach to several of the problems in lapping, edging and finishing was developed by Galvin Manufacturing Corp. by an adaptation of the familiar ball mill technique.[26] A large number of blanks (as many as 1000 in a two-quart jar) previously lapped by conventional methods such as those described above, are sorted into groups on the basis of thickness, or frequency, and each group placed in a jar containing a mixture of abrasives. A motor driven device such as the one shown in Fig. 15 may be used to rotate several jars at a time.[27] The usual speeds are from 35 to 60 r.p.m. Higher speeds may cause excessive breakage.

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[25] Manufactured by Sipp-Eastwood Corp., Optical Division, Paterson, N. J.

[26] Fruth, Hal F., Crystal finishing: *Radio-Electronic Engineering* (*Radio News*), **3**, 3–7, 26, 44–45 (1944).

A two-quart glass jar, 6″ in diameter with four straight sides, rotated in a horizontal position was found to give the best tumbling and mixing action for the abrasive. Cylindrical jars do not turn over the crystals and the position of baffles placed in cylindrical jars to accomplish this are critical and not convenient. Devices for rotating the axis of rotation of the jar as well as the jar itself have been used. Various combinations and types of abrasives have been used for the milling methods. A jar about one-half to five-eighths filled with solids and the remainder with water containing a wetting agent was found to give good results. Two types of abrasives are used simultaneously in the mill. #320 to #800 silicon carbide or aluminum oxide comprise approximately one-tenth by volume of the

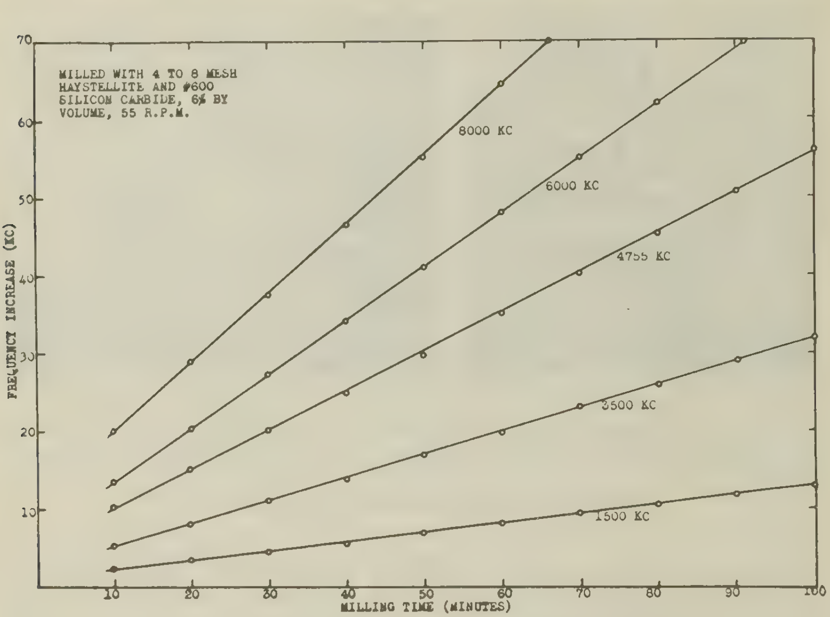

FIG. 16. Frequency increase with milling time. (Courtesy of Galvin Mfg. Corp., Chicago, Ill.)

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[27] Manufactured by Jenkins Engineering Co., 2301 East 48th Street Terrace, Kansas City 4, Mo.

solids and do the grinding while a dense material such as garnet or corundum in the form of pebbles (5–10 mesh) supplies the pressure. The amount of quartz removed is linearly related to milling time and due to the extremely small pressures, the method is much slower than the usual lapping techniques. The rate of frequency increase will depend among other factors on the frequency-thickness curve and the starting frequency as shown in Fig. 16. When such factors as velocity of rotation, shape of container, type of abrasives, etc., are controlled the results are reproducible within very close limits. The method does not equalize thickness differences. In practice, the crystals are channelled (frequency grouped) within very close limits if a small frequency spread is desired at the end of milling. If this procedure is not followed, all the crystals are removed from the mill at stated intervals and channelled. Those that are within the frequency tolerance are removed and those below the specified range are returned to the mill for further milling and the process repeated. An alternative procedure is to start the mill with the group of crystals which is furthest away from the final desired frequency and then to add the channelled groups one at a time in progressively increasing frequency of the next channelled group.

The method is used to increase the frequency only a small amount, approximately a few hundred KC being the maximum for a 6 MC BT-cut while 25 KC is the usual amount removed. The rate of removal of quartz is very small and the contour and edges of the crystals may seriously deteriorate if milled for long periods. The method is said to be subject to such control that most of the abrasion is on the major surfaces of the blanks, or if desired, the edges can be made to abrade relatively more rapidly.

The method has been employed by several crystal plants and has been most successful where large production quantities of closely spaced frequency channels are required as in the present crisis. The mass etching procedures which are now used as standard finishing practice and the high speed precision lapping techniques available today are capable of huge precision-controlled production so that it is difficult to evaluate its importance as a technique that will be useful in the future. A possible application of the method is in the manufacture and final frequency adjustment of very thin crystals. The method has been useful in edging and washing crystals.

FINAL FREQUENCY ADJUSTMENT OF QUARTZ OSCILLATOR-PLATES

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ABSTRACT

The art of finishing machine lapped quartz oscillator-plates to final frequency and activity specifications is discussed. Two general methods have been practiced: the abrasive lapping technique, now abandoned because of undesirable ageing phenomena caused in the plate, and the etching-to-frequency method. The *x*-ray irradiation technique has a special field of application in making highly precise frequency adjustments and in salvaging over-frequency plates. The cause and cure of frequency and activity ageing in quartz plates is discussed, and methods of adjusting the frequency of metal plated wire-suspension type plates are described.

INTRODUCTION

The machine lapping of AT and BT quartz oscillator-plates is usually stopped somewhat over the final desired thickness. The final finishing operation as ordinarily applied involves a process of lapping or etching the individual crystals by hand, with intermittent electronic measurement of the accompanying change in frequency, until the final desired frequency is reached. Control over the thickness of the crystal cannot be had at this stage by mechanical measurement since the dimensions involved are of the order of millionths of an inch. At the end of the finishing operation the crystal is cleaned and assembled in its permanent holder. The unit is now tested over a specified range of temperature, say from −55° to +90° C., to see if the frequency and activity remain within prescribed tolerance values, and then passes through various mechanical tests and inspections before it is submitted to Government inspectors for final acceptance.

FINISHING BY THE ABRASION METHOD

In this method the crystals are first lapped by machine until they are within roughly 0.04 to 0.4% of the final desired frequency. The plates are then lapped individually by hand with fine abrasive on a glass plate. The crystal frequency is measured from time to time during the operation, usually in a comparison oscillator against a crystal standard of the desired frequency, and the duration of the hand lapping is gauged accordingly. The crystal should be measured in the particular pair of electrodes

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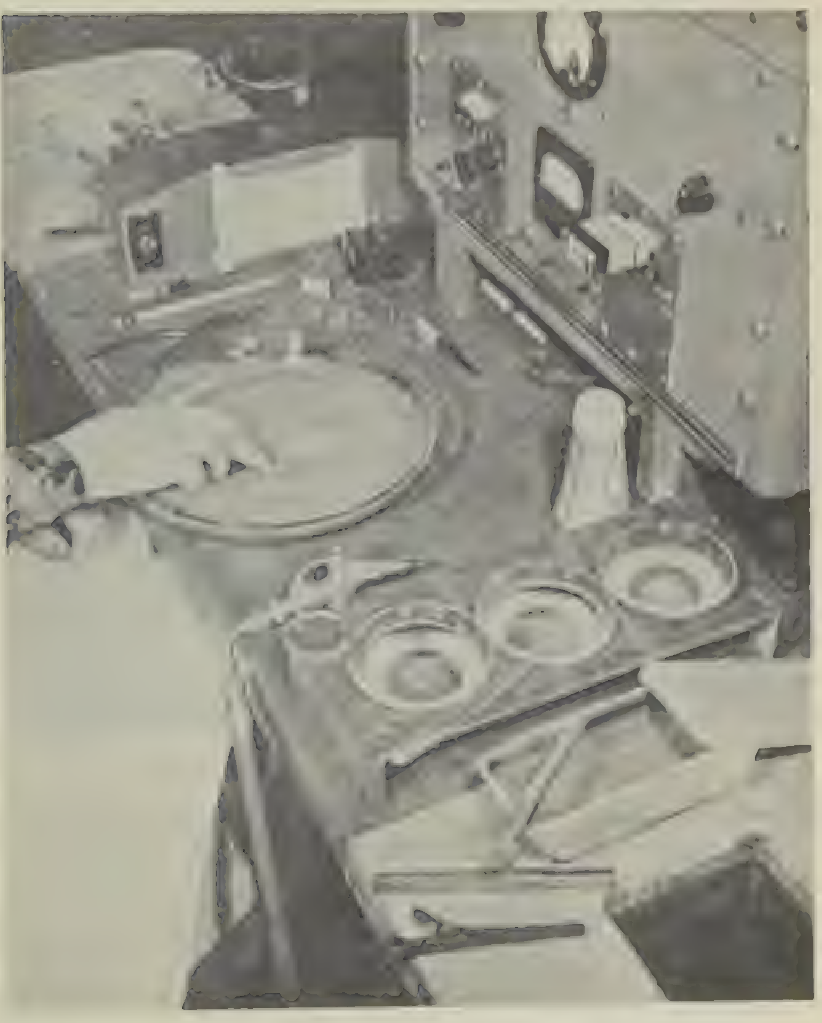

FIG. 1. Final abrasive finishing position, showing glass lap (held in a pie dish) with crystal and abrasive, comparison oscillator with standard crystal, polariscope (left) for determining X and Z' directions, sink with soap solution and tooth brush for cleaning, air gun for drying, and lead dish of etching solution with plastic holding tongs for acid wash.

in which it is to be permanently mounted. It is highly desirable to use a relatively fine emery abrasive, preferably 303½, or a fine garnet finishing powder in the final lapping. The operator lightly holds the plate on two opposite corners by the finger tips. The glass lap plate is then circled with a figure-eight or epicyclic motion and the fingers are then transferred to the opposite corners. This process is repeated on both sides of the crystal. By suitably holding the crystal a skilled operator can control convexity or wedging in the plate. Generally speaking, it is desirable to finish high frequency BT plates so that they are slightly convex, the centers being higher by a few hundred thousandths of an inch, since this condition is found to promote crystal activity. Very flat plates or wedged plates usually have relatively low activity and concavity usually proves fatal. The contour can be examined by wringing down on an optical flat and observing the system of interference fringes or by sensitive surface gauges such as the Pratt and Whitney Electrolimit gauge. It is sometimes found convenient to lap the crystal by wringing down on a small optically flat glass dop which is then manipulated by hand over the lapping plate. Groups of crystals can be handled simultaneously in this way by wring-

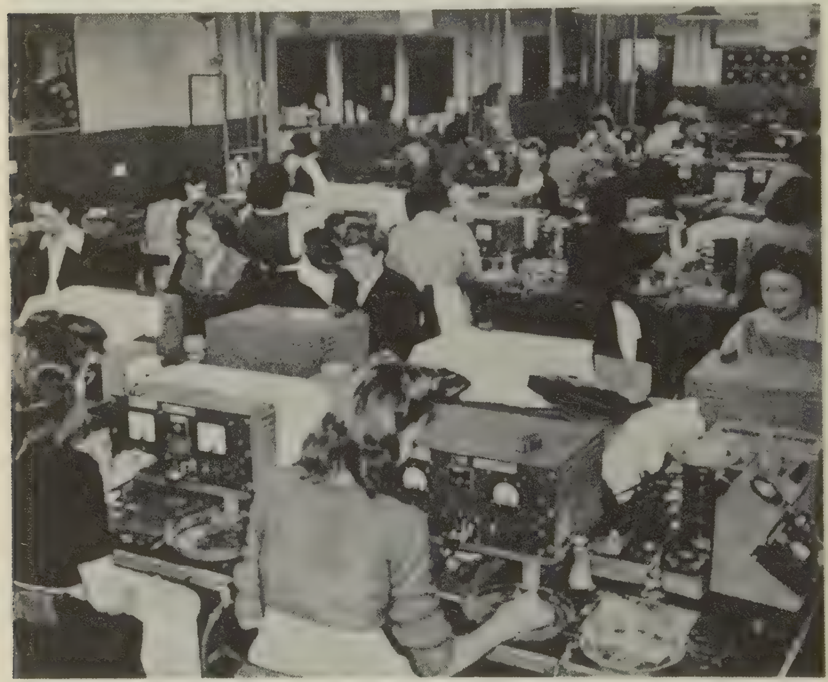

FIG. 2. View of final finishing department in a crystal plant.

ing or cementing down on a large optical flat. Needless to say, the flatness of the glass lapping plate itself must be carefully maintained.

In the finishing process attention must also be paid to the so-called activity of the crystal. The activity is primarily a function of the edge dimensions of the plate in a given electric circuit and holder, but also is influenced by the contour, the fineness of abrasive finish, cleanliness, the presence of cracks and chips on the edges, twinning, inclusions and other factors. In so-called predimensioned AT and BT plates the edge dimensions are deliberately preselected so as to give minimum coupling between the fundamental high frequency shear mode vibration and high harmonics of the Y flexural mode. Such crystals have inherent high activity and are relatively free from spurious frequencies and from activity dips over a range of temperature. Accurate control of dimensions, usually to 0.001 inch in low frequency crystals, and of cutting angle, with both the ZZ' and XX' angles held to at least ±15 minutes of arc, is essential. It is general practice, however, especially in the higher frequencies, to cut all crystals during manufacture to some abritrary set of dimensions. This is done because the ideal dimensions for a particular frequency usually are not known or conveniently determined, and because the dimensional and angular control involved is not easily achieved on a mass production basis. When arbitrarily dimensioned crystals are hand finished it is often necessary to bevel or grind down the edges of the plate in order to bring up activity. This changes the edge dimensions to a more favorable ratio.[1] The edging operation is variously done by lapping with abrasive on glass, or on a carborundum stone, fine emery paper, crocus cloth or a diamond wheel. Rotating hollow cylinders lined with diamond impregnated bakelite also are useful. Grinding the X edge of the plate is advantageous since this dimension more directly controls the interfering flexural mode. Unless the edge dimensioning of the plates preliminary to finishing is carefully done, a slight beveling and rounding of the sides and corners of the plate is desirable in order to remove roughness and chips. Rough edges and sharp corners are objectionable because of the danger of small particles of quartz becoming dislodged and wedging between the plate and electrodes.

After the plate has been brought to specifications it is scrubbed with a toothbrush and soap, thoroughly rinsed and dried, rechecked for frequency and activity and then mounted permanently in its holder. The holder and electrodes must also be clean. Drying should be done with a jet of clean dry compressed air. Rubbing with a towel is objectionable because the surface of the plate becomes loaded with cellulose and the plate frequency is decreased thereby. Towelled plates when baked at *ca.* 500° C. gain exactly the same amount of frequency lost by the towelling operation, presumably due to burning off of the organic matter. Slight upward or downward changes in frequency can be effected by changing to electrodes with a higher or lower air gap, respectively, and a further downward change in frequency can be effected by the *x*-ray method described beyond. Plates can be given a crude partial temperature test during the finishing operation by heating them during measurement over an alcohol burner or electric stove or by cooling with a piece of dry ice. Activity dips recognized in this way can be removed by edge grinding, the changes in the plate dimension moving the dip to higher temperatures until it goes beyond the upper temperature limit.

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[1] Temperature test rejects in randomly dimensioned plates usually run between 15 and 40 per cent. The rejected crystals are disassembled and run through the finishing operation again, where the edge dimensions are changed to a more favorable ratio. Temperature test reject rates in predimensioned crystals ordinarily are only a few per cent.

Various mechanical lapping contrivances have been devised to speed up the initial stages of the finishing operation. The Sipp-Eastwood or Seco lap comprises two rotating lapping discs with a thin perforated workholder containing a single quartz plate. The workholder, ordinarily made of vinylite plastic or zinc, is held in position by a metal post and has a rotatory motion imparted by the lapping discs. The Empire Electronics Co. lap is a small scale version of the drill press type of mechanical lap. These laps are described in an accompanying paper.[2]

Electrodes and Springs. Most of the high frequency crystals currently in mass production, notably the CR-1 and FT-243, are clamped between stainless steel electrodes about 0.050 to 0.060 inches thick. The corners of the electrodes are raised somewhat, giving an air gap which usually ranges between 0.0004 and 0.0008 inches. Maximum activity is obtained in the lower gaps and the plate will ultimately refuse to oscillate as the air gap is increased. Both the lands and the air gap areas must be flat and parallel to at least within 0.0001 inches. This is necessary in order that the crystal withstand drop and vibration tests and to avoid certain undesirable modes of vibration which result in activity and frequency failures in the temperature testing. The electrodes are held in place by a spring exerting a force of about 5 to 8 pounds. Excessive spring pressure may damp out undesirable subsidiary modes of vibration but tends to reduce activity, while low spring pressure may result in failure in the drop and vibration tests.

Care should be taken in low frequency AT clamped plates to avoid particular air gap dimensions. Acoustic waves generated by the oscillator-plate are reflected back from the electrode and at certain gaps resonance will occur resulting in damping and diminished activity of the crystal. The air gap dimensions to be avoided are given by

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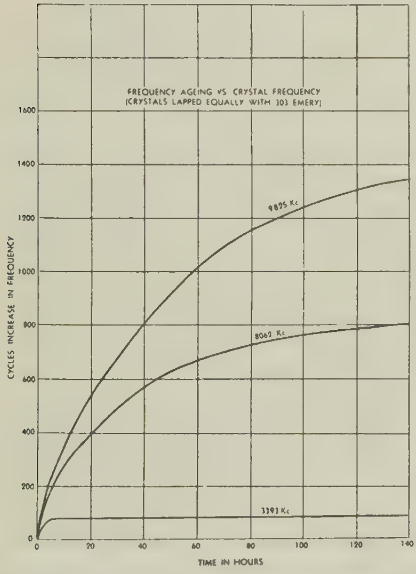
[2] Parrish, W.: Machine Lapping of Quartz Oscillator-Plates, *Am. Mineral.* this issue.

$$l = \frac{pv}{2f}$$

where l is the air gap in inches, v the velocity of acoustic waves in air at room temperature and pressure (=1083 ft/sec.), f the plate frequency in cycles per second, and $p = 1, 2, 3. \ldots$ The effect is of little or no consequence in high frequency BT plates.



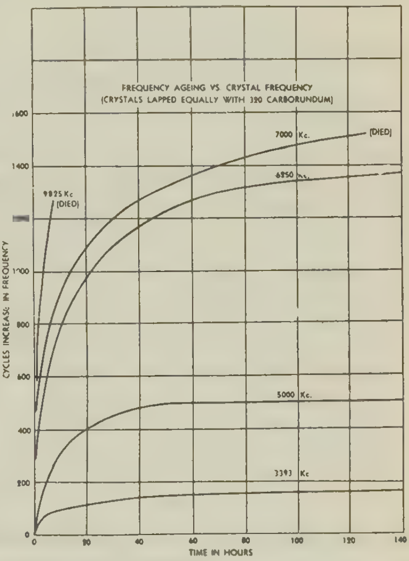


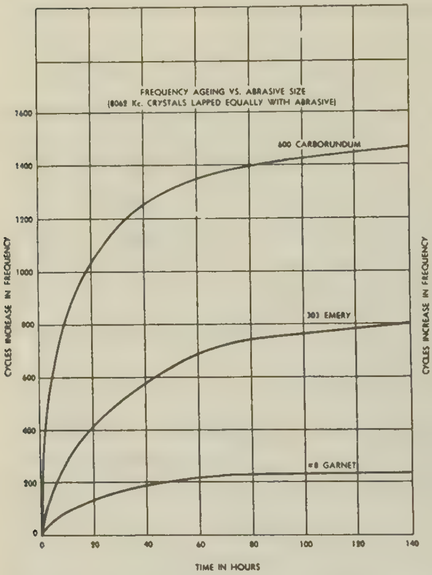
FIG. 3. Relation of frequency ageing to crystal frequency. All are BT crystals lapped identically with 303 emery.

FIG. 4. Relation of frequency ageing to crystal frequency. All are BT crystals lapped identically with 320 carborundum.

AGEING PHENOMENA IN ABRASIVE-LAPPED PLATES

The Armed Services have had serious difficulties with abrasive-finished high frequency crystals due to deterioration in storage after acceptance from the manufacturer. The ageing consists of a spontaneous increase in frequency, often to such a degree that the upper frequency tolerance is exceeded. There is an accompanying decrease in activity, usually at a slower rate, and this may continue until the plate will no longer oscillate in the test circuit. Probably several million high frequency crystals which initially passed all manufacturers and Signal Corps tests were lost by ageing before the cause and cure of the effect were found. Most of these crystals have since been salvaged. The principal features of quartz ageing are as follows:

1. In a series of plates of different frequencies that have been lapped identically with abrasive the amount of frequency ageing increases with increasing plate frequency. (Figs. 3 and 4.)
2. In a series of plates of the same frequency the frequency effect is more marked the coarser the abrasive used (Fig. 5).



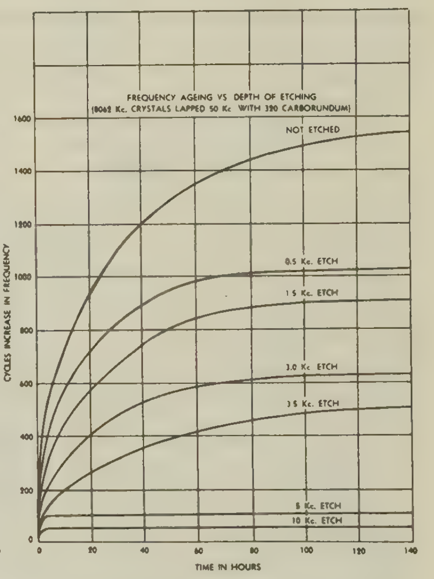


Fig. 5. Relation of frequency ageing to abrasive size. 8062 Kc BT crystals lapped 50 Kc by hand with abrasive of the type indicated.

Fig. 6. 8062 Kc crystals lapped 50 Kc with 320 carborundum and then etched varying amounts. The frequency ageing decreases with increased depth of etching up to a limit of roughly 0.5 micron of quartz.

3. In a group of plates lapped identically with abrasive the frequency ageing is both accelerated and accentuated if the plates are kept wet. The reverse is true if the plates are kept dry over a powerful desiccant. Immersion in various organic liquids has little effect on the ageing and no correlation is found with the dielectric constants or other properties of the liquids.
4. Abrasive lapped plates allowed to age over a lengthy period are found on examination to have a loose dust-like deposit of quartz over their surface. Further, the plate may be covered by a thin adherent film of silica that can be scraped up by a pointed medium. This film may be thick enough to visibly reduce the air gap between clamped electrodes; when the plate is rubbed with a towel the air gap increases, and the frequency may increase as much as several kilocycles over the original value. The film consists of hydrated (?) silica together with remnants of finger grease, abrasive, and soap or other agents used in cleaning.

5. Activity ageing is more marked and more rapid the coarser the abrasive finish and the higher the plate frequency. Generally speaking, the decrease in activity with time in crystals lapped with 303 emery or finer abrasive is measurable but does not commonly exceed tolerances in frequencies below about 6500 Kc. The deterioration becomes more serious with increasing frequency and the great majority of abrasive finished, unetched, crystals over about 7500 to 8000 Kc either drop below activity tolerance or go dead entirely with time. In a series of BT crystals finished with 320 carborundum it was found that in frequencies from 3400 to 5000 Kc many of the crystals still oscillated, with diminished activity, after standing five months, but that over about 6000 Kc all crystals soon went dead. In a series of 9825 Kc crystals, those finished 50 Kc with #8 garnet were still weakly active after five months while those finished with 600 or 320 carborundum went dead after periods of a few hours up to a few weeks.

The principal cause of ageing is believed to be a deterioration of the surface of the quartz oscillator-plate itself. Due to the abrasive action in lapping the surface of the plate becomes strained and cracked on a submicroscopic scale. This thin surface skin of misaligned quartz tends to spall off or recrystallize with time, aided apparently by adsorbed and capillary water, and the increase in frequency reflects the decrease in effective thickness of the plate. Direct evidence of the damaged surface layer has been obtained by *x*-ray, electron diffraction, and electron microscope studies; this work, done by others, will be published and need not be described here.[3] Other effects which may also contribute to undesirable frequency and activity changes with time are fatigue and corrosion of the springs and other metal parts of the holder, absorption of moisture, and deterioration of the phenolic material composing the body of the holder itself. These result in loading of the crystal by water or organic material and in decreased spring pressure.

It is found that ageing effects can be eliminated if the oscillator-plate is etched in a solvent as the last step in manufacture, thus removing the strained and misaligned material and exposing solid bedrock. Data illustrating the decrease in frequency ageing accompanying various depth of etching are shown in Fig. 6. This discovery led the Signal Corps to specify that all high frequency crystals must be etched instead of abrasive lapped to frequency. The minimum etch specifies one micron of quartz and contains a safety factor of roughly 2 to 3.

FINISHING BY THE ETCH METHOD

In the etching-to-frequency technique, the machine lapping is stopped when the crystals are a little more than one micron above final thickness. The crystals are now etched in bulk, in a single continuous operation, to remove one micron of quartz—the minimum Signal Corps requirement.

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[3] See also Bottom, V., Ageing of Quartz Crystal Units, Camp Coles Signal Labs., Crystal Branch, Eng. Memo, No. 4, 13 pp., June 29, 1944.

In this operation, the crystals may be racked together in notched copper, plastic or Pyrex trays (Fig. 7), or positioned on endless chains or suspended baskets, and then immersed for a predetermined time in a tank or shallow dish of etching solution. The solution used in mass etching usually is saturated with NH_4HF_2 and is kept at a temperature of 40 to 50° C. The amount of etching is ordinarily so controlled as to bring the crystals to about 0.02 to 0.08 per cent less than final frequency. The crystals are now issued to the finishing operators who etch them individually in a small bowl of solvent, with accompanying measurement of frequency, until the desired frequency is reached. The solution used in final

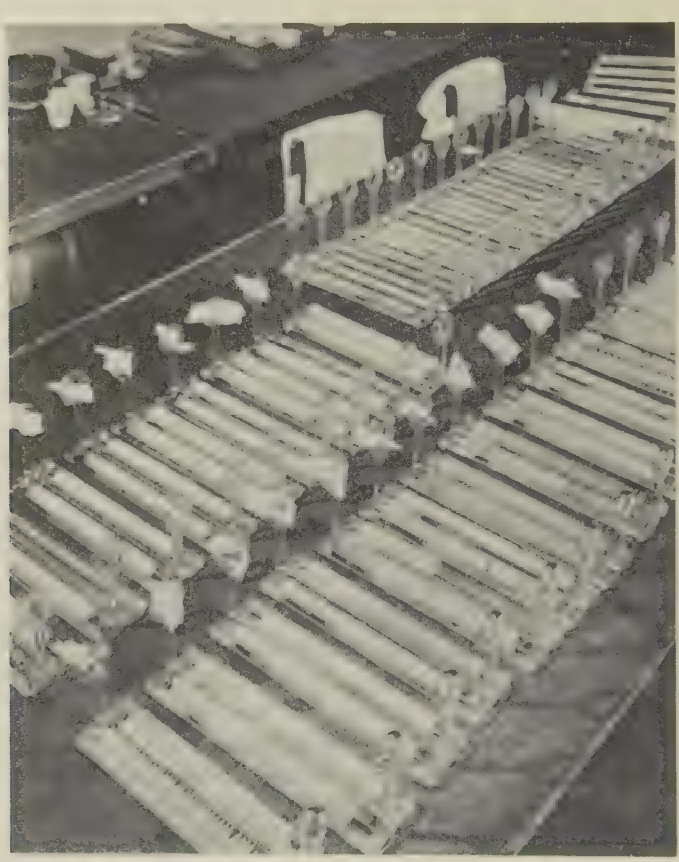

FIG. 7. Crystals racked in notched Pyrex trays, preliminary to being transferred successively to shallow tanks containing sulfuric-chromic acid cleaning solution, running rinse-water, etching solution, and running water for final rinsing.

finishing usually contains about 15 to 40 per cent by weight of NH_4HF_2 and is kept at room temperature. Hot concentrated solutions are sometimes used but small frequency changes are not then easily controlled. A typical finishing position is shown in Fig. 8. After etching, the crystals are washed, dried, and mounted in holders. If the activity drops to a low value during final etching the crystal may be edge lapped on a carborundum stone or rotating abrasive cylinder (Fig. 8). Cleanliness is essential to ensure uniformity of etching. Machine lapped crystals should be cleaned before mass etching first in organic solvents or soap and water to remove most of the lapping vehicle and carborundum and then immersed in hot solutions of trisodium phosphate plus "soapless soap." Washing before the final finishing etch also is advisable, especially if the crystals have been handled; and a final scrubbing with toothbrush and soap followed by thorough rinsing in warm running water is requisite.

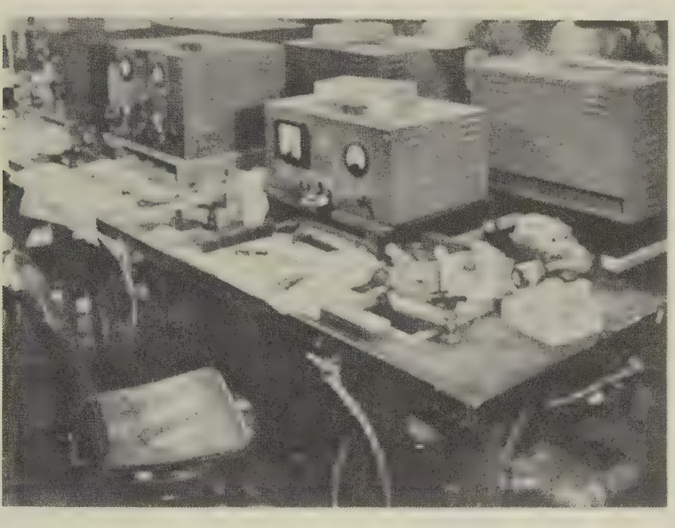

FIG. 8. Final etch finishing position. Note plastic tongs for immersing crystal in Pyrex bowl of etch solution, toothbrush and bowl of soap solution for cleaning, air gun, bowl of dilute ammonia water, comparison oscillator, and abrasive lined cylinder for beveling crystal edges.

Notes on the Etching Process. The rate of change of frequency during etching is a function of the initial frequency of the plate and increases with increasing frequency according to the frequency-thickness relation. The rate of etching is very much faster in the first minute or so than thereafter (cf. Fig. 12). This is due to the relatively rapid solution of projecting edges and corners on the lapped surfaces and is primarily an unloading effect. The effect is especially marked in dilute solutions. In crystals of the same frequency the rate of etching is faster the coarser the abrasive finish on the crystal (Fig. 9). The rate of etching also increases with increasing temperature of the etching solution (Fig. 10), and with increasing concentration (Figs. 11 and 12). Agitating the solution speeds the action. Crystals from which 5 to 10 microns or so of quartz have been etched, depending on the kind of abrasive finish, become transparent with a glazed appearance. Prolonged etching commonly results in decrease of activity due partly to relatively deep etch pits, enlargement of surface flaws, and change of contour. Different oscillator-cuts change frequency at different rates during etching because of the vectoriality of solution rates in quartz and in part, because of the differences in modes of vibration.

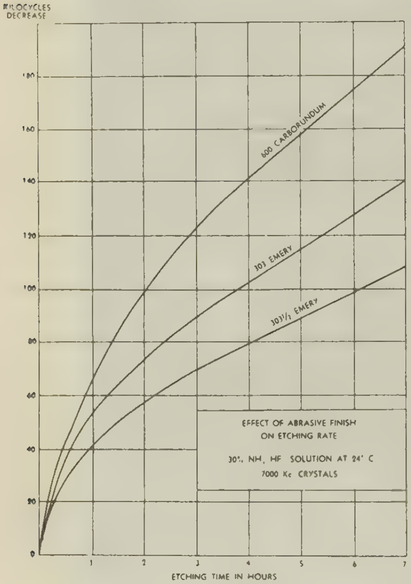

Fig. 9

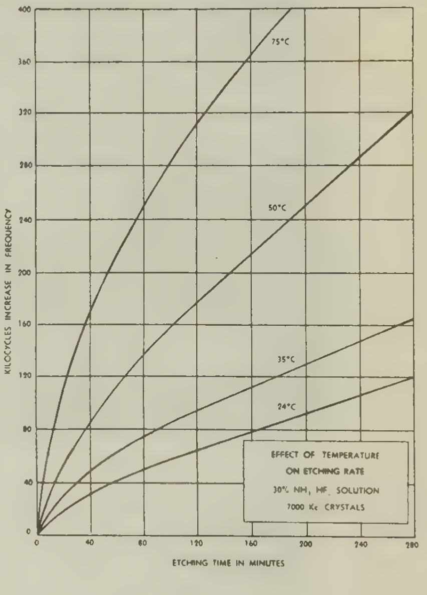

Fig. 10

Hydrofluoric acid formerly was used as the etching medium but this dangerous compound was later replaced by ammonium bifluoride and by proprietary compounds[4] based on ammonium bifluoride but containing additional ingredients to improve the quality of the work. General speaking, hydrofluoric acid gives a coarse deeply pitted effect while the ammonium and alkali bifluorides, especially when containing fluoborates, give a fine-grained even effect. Care should be taken to maintain constancy of concentration and temperature of etching solutions in order not to lose the calibration of etching rate. The solutions should be neutralized with soda ash before they are discarded to prevent damage to the plumbing.

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[4] "Safe-T-Etch" and "Frequency-Etch," sold by the Hudson American Corp., 25 West 43rd Street, New York, N. Y.

FINISHING BY THE *X*-RAY IRRADIATION TECHNIQUE

A new method for making a precise final adjustment of frequency, still under development and not yet widely used in the crystal industry, also may be mentioned. When quartz oscillator-plates are irradiated with a broad beam of *x*-rays the quartz gradually becomes smoky in color and the elastic constants are concomitantly altered in such way as to reduce the oscillation frequency. The general nature of the effect is described in an accompanying paper.[5] Briefly stated: (1) the total change of frequency that can be effected in BT plates is limited to roughly 0.006 to 0.12 per cent of the nominal frequency with an average downward change of approximately 0.02 per cent; (2) the total response varies in different specimens of quartz, for reasons not known, between the limits stated; (3) the color of irradiated plates can be discharged and the frequency restored to the original value by baking at temperatures over about 180° C.; (4) the rate of change of frequency during irradiation depends primarily on the wave-length and intensity of the *x*-ray beam

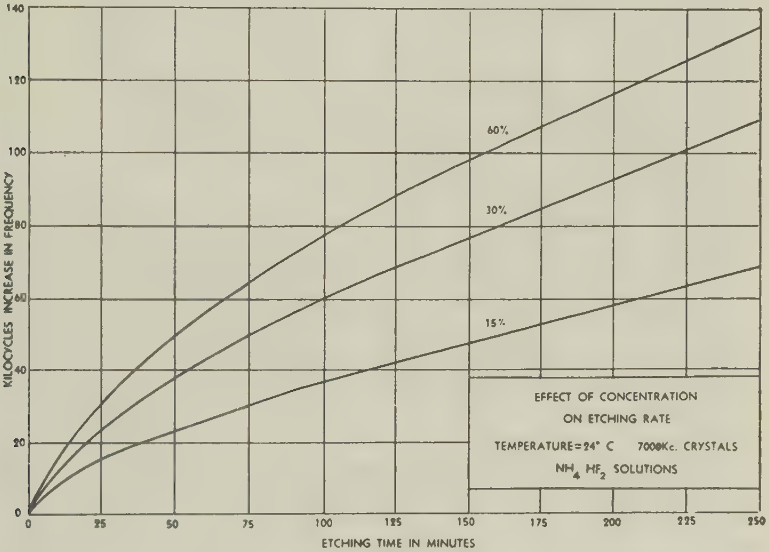

FIG. 11

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[5] Frondel, C., Effect of Radiation on the Elasticity of Quartz, *Amer. Min.*, this issue.

employed, the anode to plate distance, the thickness of the plate being irradiated, and on the total response inherent in the particular plate being treated.

The irradiation technique offers a number of important practical advantages. The frequency of the plate can be exactly adjusted under continuous visual control by oscillating it in the *x*-ray beam until it reaches the desired frequency. The downward change in frequency permits the salvage of plates that have gone over the upper frequency tolerance due to ageing or to over-finishing during manufacture. Further, surface stabilized or metal-plated crystals can be adjusted in frequency without disturbing the surface condition of the quartz; and plates can be finished to have a desired frequency at a specific temperature by irradiation at that temperature. The technique is of special advantage in the manufacture of ultra high frequency plates with tight frequency tolerances. The principal factors which limit the application of the method are the relatively long irradiation time needed with present equipment when

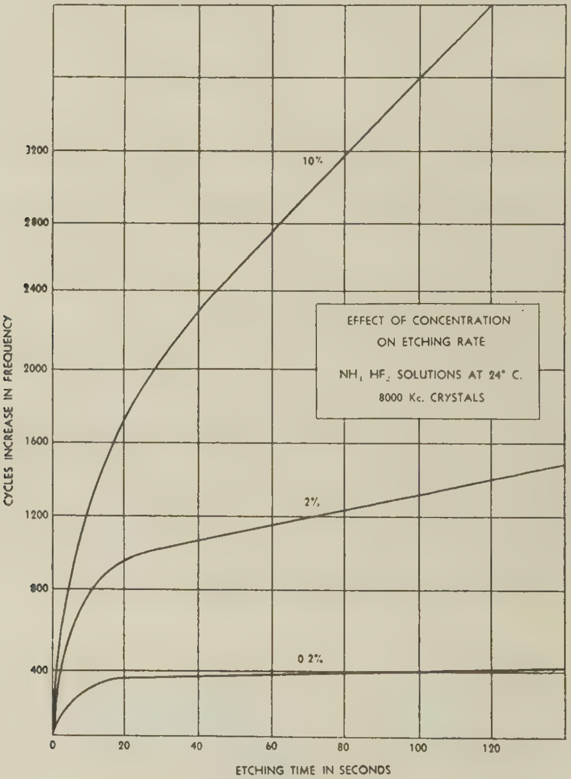

Fig. 12

large frequency changes are desired, and the high initial cost of installing sufficient equipment to handle a large volume of work. Generally speaking, the crystals have to be finished by conventional methods to within roughly 0.02 per cent of nominal frequency before irradiation since the total frequency change that can be effected is of this order.

Commercial x-ray equipment for irradiating oscillator-plates has been marketed by the North American Philips X-Ray Corporation. A photograph of three Philips irradiation units used on the production line at one of the quartz plants of Reeves Sound Laboratories in New York City is shown in Fig. 13. The equipment comprises a broad focus copper anode tube designed to operate continuously at 25 ma and 60 KV. The water-cooled tube is housed in a casting to which are attached two insulated jigs opposite the windows of the tube. The jigs consist of a rotatable shielded disc containing six variously sized electrode positions into which oscillator-plates may be inserted and in turn rotated into the x-ray beam (Fig. 14). The outer electrode facing the window is pierced with a large hole to permit the beam to strike the central area of the crystal. The jig is so constructed that the plate can be oscillated during irradiation and

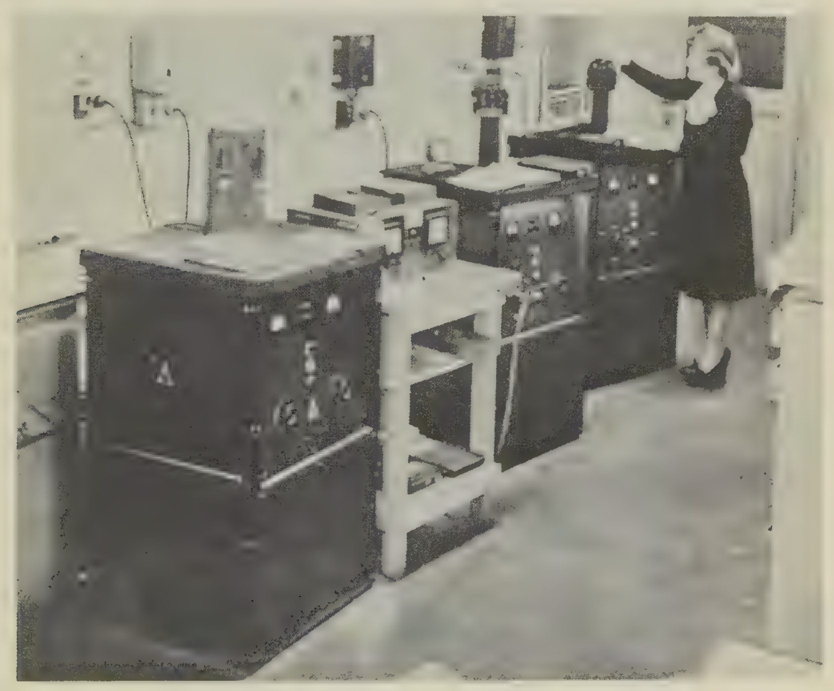

FIG. 13. Philips x-ray irradiation units used to adjust the frequency of finished quartz oscillator-plates

the frequency change followed visually on a meter. The average rate of downward change of frequency in this equipment in high frequency BT plates averages between 20 and 40 cycles a minute. Crystals mounted by wire suspension in bakelite or other holders relatively transparent to x-rays can be oscillated and irradiated to frequency directly through the holder.

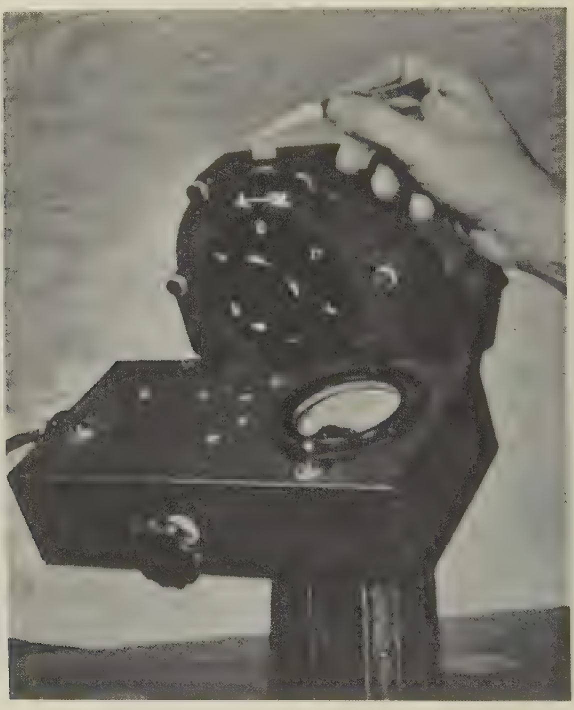

FIG. 14. Close-up view of jig used to hold and oscillate crystals during irradiation.

FREQUENCY ADJUSTMENT OF METAL PLATED CRYSTALS

A number of types of crystals are plated on the surface with a thin film of metal, usually gold, silver or aluminum. In crystals whose frequency is determined by edge dimensions, as in the GT, CT, DT cuts and X-cut bars, the frequency is relatively insensitive to the amount of metal deposited and to the thickness dimension of the quartz plate itself. Minor frequency adjustments of such crystals are effected by edge grinding, leaving the metallized surface untouched. These crystals ordinarily are mounted by clamping electrodes to or soldering wires to the nodal points. In plated AT and BT shear mode crystals, however, the frequency decreases with the amount of metal deposited and unless the plating operation itself is very carefully calibrated minor frequency adjustments are necessary. These are effected by operating on the metal plated surface itself. Shear mode plated crystals are preferably mounted by wire suspension, the wires being soldered to opposite and opposing edges or corners of the plate.

In the manufacture of plated AT and BT crystals, the plate is lapped and etched a number of kilocycles over the final desired frequency and then metal is superdeposited by cathode sputtering, evaporation, chemical deposition or ceramic processes until the frequency is brought back close to the desired value. If a further downward change is then required, a metal such as nickel can be electroplated upon the base coat or minor adjustments can be effected by *x*-ray irradiation. If an upward adjustment is desired the metal film can be carefully abraded with a rubber ink eraser or the film can be partly etched off with a solvent. Metal also can be deplated electrochemically. These adjustments can be made after the crystal has been soldered to its supporting wires. Efforts to remove large amounts of metal often result in a loosening of the film and if a large upward frequency is desired it is best to dissolve off all the metal and start over. Methods recently have been devised in which the plate can be oscillated and measured during the metal plating operation and brought directly to frequency. It is desirable to bake plated crystals at *ca.* 550° C. to bring about an increased particle size in the film and enhance its strength and adherence.

EFFECT OF RADIATION ON THE ELASTICITY OF QUARTZ

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Abstract

When quartz is exposed to x-rays, gamma-rays, electron beams, alpha-particles or deuterons it becomes smoky in color, the rate of solution in hydrofluoric acid is decreased, and the elastic constants are altered. The effect of radiation on the elastic properties is revealed by study of irradiated quartz oscillator-plates. In general the oscillation frequency decreases continuously during irradiation to a limiting value, and types of plates dependent on different elastic constants are affected unequally. The effect is being utilized in manufacture to make the final frequency adjustment of oscillator-plates.

The degree of response in color and elasticity is limited and varies among different specimens. Baking at temperatures over about 180°C. restores the original properties, and conditions the response of the quartz on re-irradiation. Luminescence phenomena accompany both irradiation and baking and, together with the response in color and elasticity, are related to the original color and composition of the quartz.

Introduction

It has been known for many years that quartz becomes smoky in color when exposed to x-rays, radium radiations, or cathode-rays (electrons.)[1] Other radiations, both of the wave-type and streams of material particles, also are effective. A photograph of the smoky color produced by a narrow beam of x-rays in a sawn block of quartz is shown in Fig. 1. Recently the writer has found that the change in color is accompanied by variation in the elastic constants of the quartz as well. This is shown by a change in the frequency of oscillation of quartz plates that have been exposed to and colored by radiation.

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* Department of Mineralogy, Harvard University, Cambridge, Mass. (on leave).

[1] Villard, P., *Compt. rend.*, **126**, 1564 (1898). Georgiewski, N., *Ann. Physik, Beibl.*, **28,** 1015 (1904). Salomonsen, C. J., and Dreyer, G., *Compt. rend.*, **139**, 533 (1904); **138,** 1543 (1904). Egoroff, N., *Compt. rend.*, **140**, 1027 (1905). Bertholet, D., *Compt. rend.*, **143,** 477 (1906); **145,** 818 (1907). Philips, C. E. S., *Nature*, **77,** 535 (1908). Goldschmidt, V. M., *Forh. Vidensk. Seleskr., Oslo*, No. 5, 1 (1906). Brauns, R., *Cbl. Min.*, 721 (**1909**). Doelter, C., *Das Radium und die Farben*, Dresden (**1910**). Newbery, E., and Lupton, H., *Mem. Proc. Manchester Lit. Phil. Soc.*, **62,** 1 (1918). Doelter, C., and Sirk, H., *Sitzber. Ak. Wiss. Wien, Math.-nat. Kl.*, **119,** 1091 (1919). Doelter, C., *Cbl. Min.*, 321 (**1923**). Holden, E. F., *Am.*

CHARACTERISTICS OF IRRADIATED OSCILLATOR-PLATES

When a BT-cut quartz oscillator-plate is placed in a powerful beam of x-rays the frequency of oscillation is found to decrease. The frequency change is rapid at first but then slows down and finally approaches a point beyond which there is no further change on continued irradiation. The type of curve obtained by plotting frequency change against exposure time is shown in Fig. 2. The total change of frequency that can be effected is variable and depends on a number of factors. Among these are the type of cut of the plate, the treatment given to the plate prior to irradiation, the kind of radiation employed, and the initial frequency, or thickness, of the plate itself. There also is a considerable variation in response among different specimens of raw quartz and hence between different plates of the same frequency cut therefrom. The observed variation in saturation value in 8000 Kc BT plates is roughly

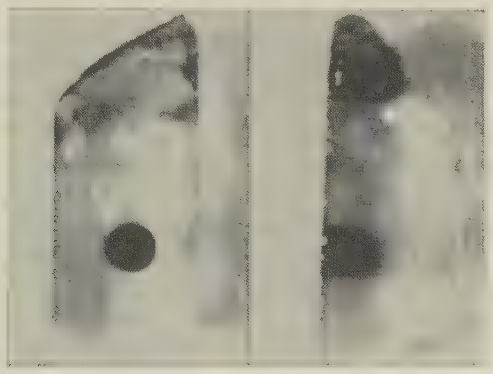
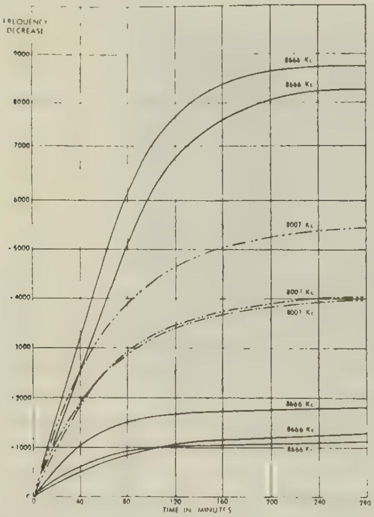


FIG. 1. Two views of a small block of quartz showing the smoky color produced by a narrow beam of x-rays.

Mineral., **9,** 1 (1924); **10,** 203 (1925). Hoffman, J., *Zeits. Anorg. Chem.*, **196,** 225 (1931); **219,** 197 (1934). Bruce, E. L., *Trans. Royal Soc. Canada*, **28,** Sec. 4, 7 (1934). Twyman, F., and Breck, F., *Nature*, **134,** 180 (1934). Mohler, N., *Am. Mineral.*, **21,** 258 (1936). Shin-Piaw Choong, *Jour. Phys. Radium*, **2,** 41 (1941). The present paper was privately issued under War Department restrictions in Nov., 1943, and July, 1944.

from 500 to 10,000 cycles decrease with an average change of approximately 1400 cycles decrease. Plates that change more than 2000 cycles are rare.

Only part of the total area of a BT crystal has to be irradiated in order to gain the maximum frequency shift. The critical area comprises roughly 30 to 50 per cent of the total area if it is circular and centrally located. Similarly, only a relatively small part of the central portion of a crystal has to be plated with metal in order to gain maximum frequency decrease by loading (Fig. 3). These observations tie in with the well known fact that the corners of shear mode rectangular plates are relatively inactive during oscillation.



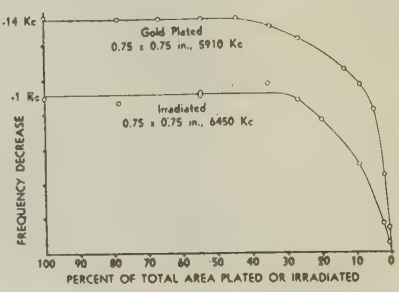


FIG. 2. Curves illustrating the variation in saturation value and rate among two groups of plates. Irradiated at 45 KV, 25 ma, 4 mm window to plate distance, unfiltered Cu radiation.

FIG. 3. Relation between frequency decrease and the area irradiated or gold plated.

The average saturation value is a function of the initial frequency or thickness of the plate and increases with increasing frequency. Preliminary measurements indicate that the average change in BT plates of a particular frequency can be calculated on the assumption that the frequency-thickness constant of irradiated quartz ($K = 100 \times 10^6$ cycles per second per 0.001 inch) is less by about 0.02 per cent than in ordinary quartz. The activity of the plates is usually slightly decreased by irradiation, except when there are material changes in coupling; and the piezoelectric constant is not grossly altered.[2]

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[2] Seidl, F., *Ak. Wiss. Wien, Ber.*, **142,** 2a, 467 (1933).

Different types of oscillator-plates respond differently to radiation. Both BT plates oscillating in high frequency shear and X-cut plates oscillating on the high frequency thickness (or X) mode are relatively highly responsive. Y-cut crystals oscillated on the high frequency shear mode are not responsive. AT shear mode plates are less responsive than BT plates. Different elastic constants or combinations of elastic constants are utilized in these types of cuts, and it appears that the constants are affected unequally by irradiation. This might mean that a particular bond in the interlocking SiO_4 tetrahedra composing quartz is more sensitive to radiation than others. Relative variation in the elastic constants leads to the possibility that plates containing closely coupled modes of vibration could be adjusted by irradiation to eliminate activity dips or achieve particular temperature coefficients of frequency. This is ordinarily done by control of dimensional ratios. Other more important practical applications of the irradiation technique are described in an accompanying paper.[3]

A rough idea of the magnitude of the frequency change to be obtained from different pieces of quartz can be gained from the luminescence phenomena described in a later section and from the original color of the quartz. Generally speaking, colorless quartz shows a wide variation from specimen to specimen in the degree of response in color to irradiation. Very low temperature hydrothermal quartz seems to be relatively weakly affected. Deep smoky quartz and citrine uniformly show a relatively small response. Amethyst quartz is entirely unaffected in color. If amethyst is first decolorized by baking, irradiation restores the amethystine color. Rose quartz, which contains traces of titanium in solid solution together with exsolved rutile needles, develops an extremely intense, almost black, smoky color. Chalcedony is weakly affected by x-rays and alternate bands in the mineral may become unequally colored. Opal is not affected; tridymite is deeply colored. The depth of smoky color produced in an oscillator-plate by x-ray appears to be directly proportional to the frequency change therein. Spectrographic analysis of oscillator-plates made variously from colorless and natural smoky quartz reveals no correlation between the kind or amount of foreign elements present in the quartz and the degree of response in color and frequency on irradiation. The principal foreign elements found in 16 samples of Brazilian quartz examined spectrographically, present in amounts over 0.001 per cent, were Al, Li, B, Na, K, Cu and Mn, listed in order of decreasing magnitude.

Rate of Change of Frequency. The principal factors influencing the rate of change of frequency during oscillation are the intensity of the x-ray beam, the distance of the plate from the window and anode of the x-ray tube, the thickness of the plate itself, the choice of wave-length, and the magnitude of the total frequency change that can be effected in the plate in question.

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[3] Frondel, C., Final Frequency Adjustment of Quartz Oscillator-Plates, *Am. Mineral.*, this issue.

The rate of change of frequency is found to be directly proportional to the intensity of the x-ray beam. The beam intensity itself increases as the square of the voltage and directly as the current passed. Data that illustrate these relations are given in Table 1. A 8007 Kc BT plate was irradiated for 5 minutes at each of the several beam intensities and the frequency change measured. The departure from the theoretical ratios is due to the non-linear relation between irradiation-time and frequency change. Using unfiltered copper radiation from a broad focus copper tube operated at 25 ma and 60 KV, with a crystal to window distance of approximately 1 mm., ordinary unsensitized BT 8 mc plates can be changed in frequency on the average about 40 cycles a minute, calculated on the basis of the initial essentially linear part of the frequency-time curves. Further data are given in Fig. 4.

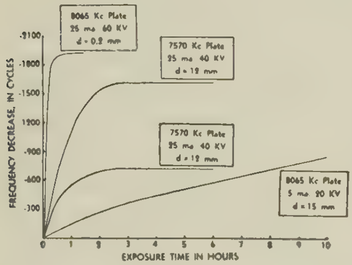
TABLE 1

	KV:	12.5	25	50	50	50	50
	MA:	25	25	25	6.25	12.5	25
Frequency Decrease in 5 minutes, in cycles		30	120	450	140	270	450
Ratio		1	4	15	1	1.9	3.2

The distance of the plate from the window and anode of the x-ray tube is an important factor, due to operation of the inverse square law. Broadly speaking, a given frequency change produced in a few minutes when the plate is 0.5 mm. from the window will require an exposure time of hours when the plate is 20 mm. distant and an exposure of many days at a distance of a foot. Measurements that further illustrate this effect are given in Fig. 5. These curves were all obtained on the same plate which was irradiated successively on the various distances cited, with the x-ray intensity kept constant.

It has already been pointed out that the average saturation value increases with increasing frequency of the plate. The average rate at which saturation is reached is also found to increase with increasing plate frequency (or decreasing plate thickness) at constant intensity of the x-ray beam. This effect is accentuated by the relatively strongly absorbed but weakly penetrating long wave-length components of the incident beam, which, while they penetrate to the same depth in a thin as in a thick plate, expose a larger percentage of the total mass of quartz as the plate thickness decreases.

The rate of change also varies with variation in the total change in frequency, or saturation value, that can be effected in a group of plates of the same nominal frequency, the experimental conditions being held constant (Fig. 2). In a group of plates of the same nominal frequency the total frequency change that can be effected varies more than twentyfold. Generally speaking, the larger the total frequency change the longer it takes to reach saturation, but no rigorous relationship appears to exist between these factors. Production data illustrating the variations in rate observed in a run of 638 BT plates of 8007 Kc frequency that had been irradiated only part of the way to saturation are shown in Fig. 6. The plates had been overshot a few hundred cycles or so during manufacture and were irradiated enough to bring them back down below the upper frequency tolerance. The frequency changes are shown on the graph as rates. These rates are the slopes of the linear portion below the knee of the frequency-time curves.



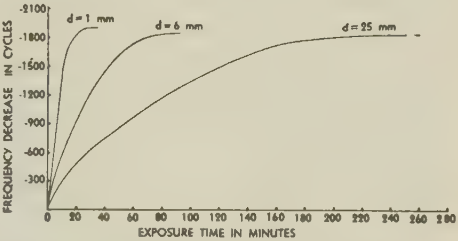
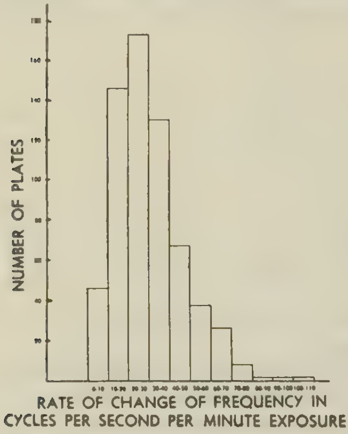


FIG. 4. Curves illustrating the variation in rate of change of frequency with x-ray beam intensity.

FIG. 5. Effect of varying window to plate distance on rate of change of frequency. 8416 Kc plate irradiated at 60 KV and 25 ma, Cu radiation.

Effect of Baking. Irradiated quartz decolorizes and reverts back to the original frequency when heated to a sufficiently high temperature. The change is a time-temperature reaction and is similar in this to the decolorization of natural smoky quartz. In the neighborhood of 180° CO a true reversal of frequency begins which is extremely slow and requires a period of weeks for completion. The rate of reversal increases rapidly with increasing temperature and over 450° the change is almost instantaneous. The reversal in frequency is accompanied both by a discharge of the smoky color and by a pale blue thermoluminescence. The increase in frequency brought about by baking is found to be exactly the same as the initial decrease brought about by irradiation. The maximum temperature at which the color of natural smoky quartz is stable is about 225° C, and the rate of decolorization, as with artificially colored crystals, increases rapidly with increasing temperature. The decolorization in this case is not accompanied by any change in frequency.



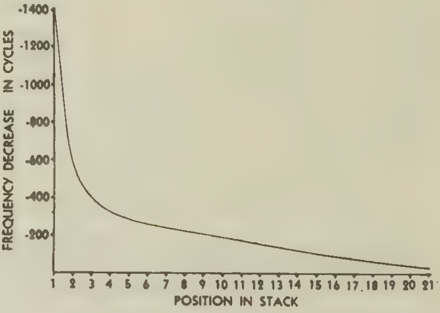


Fig. 6. Observed variation in rate of change of frequency during irradiation of 638 plates of 8007 Kc frequency under constant experimental conditions.

Fig. 7. Frequency decrease in unit time through a stack of twenty-one 8416 Kc plates irradiated at 15 ma and 45 KV with heterogeneous Cu radiation. A 8416 Kc plate is 0.01188 inches thick.

Baking and decolorizing an irradiated plate increases the rate of response of the quartz on re-irradiation. The sensitization is most marked at the limiting decolorization temperature of *ca.* 180° C. and becomes progressively less at higher temperatures until the rate equals and then becomes less than that of fresh quartz. The sensitization may involve the formation of F′ centers, as in the photolysis of the alkali halides.

Rate of Solution of Irradiated Quartz. If a quartz plate is irradiated through a lead shield pierced with a small hole, so that there is a sharply defined smoky area produced in the quartz, and the plate is then deeply etched, it is found that the irradiated area is less affected and appears on the surface of the plate as a slight eminence. In other words the rate of solution of irradiated quartz is less than in ordinary quartz. It is also found under certain not easily reproducible etching conditions that sawn and lapped sections of ordinary quartz sometimes become differentially etched along parallel bands or zones. Crystals with alternately banded smoky and colorless portions may show marked effects of this kind. The behavior reflects a local variation in the properties of the quartz itself. Oscillator-plates apparently change frequency slightly less rapidly if they have an abrasive-lapped instead of an etched surface. This effect may be due to the existence of a rough skin of quartz in the former case which does not participate in the oscillation and which has a filtering and shielding action on the incident radiation.

Absorption of X-Rays. Stacked Plates. Quartz is relatively transparent to *x*-rays and a large part of the incident radiation is transmitted through a thin oscillator-plate and does not contribute to frequency changes therein. The efficiency of utilization of the *x*-ray beam can be increased by appropriately relating the wave-length of the incident radiation to the particular thickness of the quartz plate being treated, or, alternatively, by stacking a number of plates one behind the other and irradiating all of them simultaneously. Since the mass absorption coefficient varies with wave-length, frequency changes measured in successive crystals down through a stack vary markedly from an exponential law due to the filtering action of the preceding plates in removing the softer, easily absorbed and more strongly ionizing components of the incident heterogeneous radiation. Fig. 7 shows the frequency changes effected in unit time in twenty-one 8416 Kc plates stacked together. Three identified plates were measured in each position and the results averaged. Very similar curves are obtained by measuring the absorption of *x*-rays with a Victoreen roentgen-meter in varying thicknesses of metal or quartz. Oscillator-plates can be used successfully as dosimeters or roentgen-meters in measuring or comparing the intensity and quality of *x*-ray beams and in measuring the ionizing equivalents of *x*-rays to other kinds of radiation.

LUMINESCENCE PHENOMENA

Natural quartz when heated in the dark often exhibits a pale bluish-white thermoluminescence (phosphorescence). The luminescence begins at a rather low temperature and after increasing in intensity with increasing temperature to about 300° to 400° C. gradually dies out. The intensity varies widely in different specimens of quartz, including specimens from the same locality, and sometimes is found to be confined to particular growth bands or parts of the crystal. About one half of 232 Brazilian crystals tested showed a noticeable glow on heating. Further observations have been given by Goldschmidt[4] and by Köhler and Leitmeier.[5] The statement of Ichikawa[6] that triboluminescence, a related phenomena, occurs in ordinary quartz but not the smoky varieties was not verified; the smoky color only obscures the bright internal reflection of the surface sparks seen in colorless quartz. The effect is relatively weak or absent in smoky quartz. Banded smoky sections of quartz, however, always are more affected by x-rays in the colorless regions whether these parts are thermoluminescent or not. No difference is found in the frequency-thickness constant of natural thermoluminescent and non-thermoluminescent quartz. A complex sequence of luminescence effects is obtained when natural banded smoky and colorless quartz is irradiated and heated.

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[4] Goldschmidt, V. M., *Forh. Vidensk. Selskr., Oslo*, No. 5, 1–19 (1906).

[5] Köhler, A., and Leitmeier, H., *Zeit. Krist.*, **87**, 146 (1934).

[6] Ichikawa, S., *Amer. J. Sci.*, **39**, 455 (1915).

Irradiated quartz is brightly thermoluminescent when heated. The glow is confined to the irradiated area. The luminescence begins at a lower temperature than in natural quartz, below 100° C., and the intensity is considerably greater. The glow is not accompanied by a change in frequency or in smoky color at temperatures below the 180° C. stability limit previously mentioned. The margins of the irradiated area at first glow more intensely than the interior parts. In an irradiated plate the thermoluminescence may persist after the quartz has been heated to a temperature at which the frequency and smoky color are discharged.

Quartz exhibits a bluish-white fluorescence during irradiation with x-rays. The samples also phosphoresce briefly. The intensity of the fluorescence varies considerably in different specimens. A relatively intense glow is met with in material that is not thermoluminescent and that has an original smoky color, but a number of exceptions were noted. It is usually found that the more intense the fluorescence, the more intense is the total response in frequency and in smoky color produced by the radiation, but several marked exceptions were observed. The intensity of the fluorescence apparently varies during irradiation. If a fluorescent plate is irradiated to saturation (in frequency and color) over a small area, and the plate is then moved laterally so that the incident x-rays overlap a fresh area of quartz the fluorescence in that area is much more pronounced. It may be observed in this experiment that the margin of the initially saturated area fluoresces relatively brightly. An interesting thing about the effect of x-rays on quartz is the lack of consistency in the response of different specimens. The correlation of luminescence phenomena, saturation value in frequency, rate of change of frequency and baking effects with each other, and with characters such as original color, more or less tend to follow general rules but always with marked exceptions. Part of the difficulty is in controlling the wide variety of experimental conditions.

Origin of the Luminescence and the Frequency Change. A possible origin for the change in elasticity is suggested by the luminescence phenomena. Radiations of relatively low energy content, such as ultraviolet light, when impinging on a crystal may effect a momentary transfer of electrons from lower to higher levels surrounding an atom. The infall of these electrons to their original level is accompanied by a release of energy and this appears as light in the long ultraviolet or visible parts of the spectrum (fluorescence). The displaced electrons also may be frozen in the metastable positions to be later released by thermal agitation of the structure, or otherwise, with emission of light (phosphorescence). With radiations of shorter wave-length and greater energy content as in the x-ray region of the spectrum, electrons may be completed ejected from an atom—an internal photoemission. The change may be permanent under ordinary conditions if the ejected electron is caught elsewhere in the structure, such as by an oppositely charged atom, a vacant lattice position or other type of electron-trap. "Holes" of positive potential to trap photoelectrons may exist in quartz through a Si-deficient defect structure; and atoms of a trivalent metal such as Al in substitution for Si similarly may act as electron receptors. In any case, a transfer of electric charge within the structure would result in a change of the interatomic bonding forces, and this in turn would be reflected by changes in the elastic properties and the piezoelectric oscillations dependent thereon. The limitation in the total change of frequency that can be effected presumably reflects the number of electron-traps available in the crystal. Baking the crystal restores the original properties by supplying energy to lift the trapped, metastable, electrons over their potential barrier and returning them to the atoms, now with a positive potential, from which they were originally ejected. The energy released in the infall appears as thermoluminescence. The color of natural smoky quartz appears to be a separate problem, since it is discharged at higher temperatures and is not then accompanied by a frequency change.

Pigmentation of crystals resulting from the ejection of electrons is well known, as in the blackening of silver bromide and in the production of color-centers in alkali halides and other substances.[7] A wide variety of changes in chemical systems dependent on electron transfer are similarly brought about by ultraviolet light.[8] The variation in elastic constants which is presumed to accompany these changes, however, ordinarily would tend to go unrecognized because of the minuteness of change and the insensitivity of the usual methods of measurement. Ultraviolet rays and x-rays have been found, however, to increase both the rigidity and elastic limit of NaCl crystals.[9]

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[7] Mott, N. F., and Gurney, R. W., *Electronic Processes in Ionic Crystals*, Oxford (**1940**); Rollefson, G. K., and Burton, M., *Photochemistry*, New York (**1942**).

[8] Heyroth, F. F., *The Chemical Action of Ultraviolet Rays*, New York (**1941**).

Color Distribution Phenomena

Evenly irradiated plates sometimes develop alternately light and dark smoky bands up to 1 mm or more in width. Some extreme examples are illustrated in Fig. 8. The bands are arranged parallel to the external faces of the mother crystal and are similar to the color bands commonly seen in natural smoky crystals. When natural quartz containing smoky bands is irradiated, the colorless bands become relatively deeply pigmented (Fig. 9). When banded natural quartz is decolorized by baking and then irradiated the original relatively light colored parts again are more deeply affected by the x-rays. Color bands also may develop in entirely colorless and water clear quartz. Instead of bands some specimens develop irregular or polygonal smoky areas. The color response apparently is conditioned by a sensitizing factor which was areally distributed within the crystal during growth.

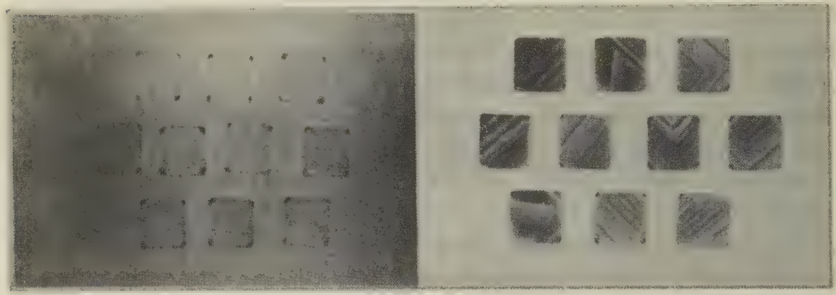

Fig. 8. Smoky bands developed in quartz oscillator-plates by x-rays. Left, before irradiation; right, after irradiation. The color bands are parallel to the rhombohedral faces.

A very remarkable type of variation in color response is found in twinned quartz. It is often observed that the two parts of a natural Dauphiné twinned quartz crystal become unequally colored when a broad beam of x-rays is allowed to fall across the twin boundary. The effect is not due to the difference in orientation in the two parts of the twin. Differential coloration is not produced in quartz that has been twinned artificially on the Dauphiné law by heat or pressure. On the other hand, irradiated differentially colored naturally twinned crystals that have been artificially detwinned by heat, and at the same time decolorized, are found on re-irradiation to become again differentially colored along what was the original twin boundary. Brazilian twins also are differentially colored by radiation. Occasionally natural smoky crystals of quartz are observed in which the color is limited against twin boundaries, and the feature is quite common in amethyst. The origin of the differential coloration of twinned quartz is problematical. The content of foreign elements in a crystal, and which could possibly act as a sensitizing factor, differs between the various form loci as shown by Frondel, Newhouse and Jarrell.[10] If, now, the twinning brought unlike faces into juxtaposition, such as the effect of Dauphiné twinning in bringing $(10\bar{1}1)$ and $(01\bar{1}1)$ faces together and coplanar, opposite sides of the twin boundary could have differences in composition and hence respond unequally to irradiation. The trouble with this theory, however, is that both Dauphiné and Brazil twins restricted to $(10\bar{1}0)$ prism faces respond unequally over the twin boundaries and in this case the two parts of the twin are chemically identical. Alternatively it can be imagined that there has been a diffusion of a sensitizing factor within the crystal which was dammed up at the

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[9] Podashevsky, M. N., *Phys. Zeits. Sowjetunion*, **8**, 81 (1935).

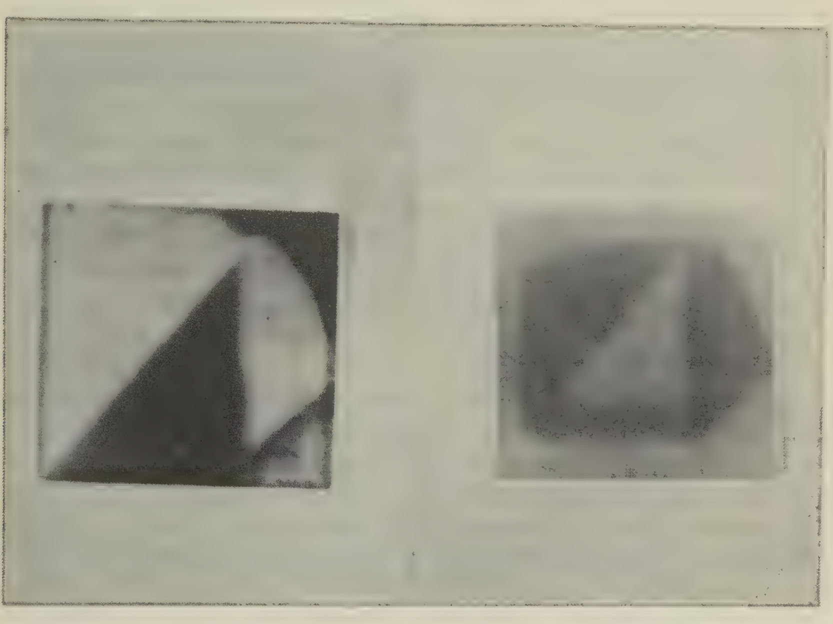

FIG. 9. Quartz plate containing natural smoky banding before (left) and after (right) irradiation. The originally colorless parts are more affected.

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[10] Frondel, C., Newhouse, W. H., and Jarrell, F. R., *Am. Mineral.*, **27**, 726 (1942).

twin boundary; an instance somewhat of this type involving the restriction of color centers to one individual of two intergrown KCl crystals is described by von Hippel.[11]

EFFECT OF RADIATIONS OTHER THAN X-RAYS

A number of different kinds of radiation have been found that cause color and frequency changes in quartz. These include both radiations of the wave type, more particularly x-rays and gamma rays, and streams of material particles including alpha-particles, electrons (both cathode rays and beta-radiation from radioactive decay) and deuterons. Deuterons, which are doubly charged nuclei of heavy hydrogen (deuterium) atoms, are highly effective. Neutrons and radiations in the wave spectrum of wave-length longer than x-rays have not been found to pigment or reduce the frequency of quartz oscillator-plates. Of the effective radiations, x-rays are the only practical choice for manufacturing operations although the radioactive radiations have in certain circumstances a definite application.

Frequency Adjustment with Radon. Radon, a gas formerly known as radium emanation, is one of the radioactive decay products proceeding from uranium and radium and ultimately ending in the 206 isotope of lead. The members of the series with the exception of the end product, lead, are unstable and transform one into the next at definite rates. The transformation is accompanied by the emission of radiation, either alpha-particles or beta-particles plus gamma rays. Radon itself emits only alpha-particles but the freshly prepared gas progressively disintegrates into RaA, RaB, etc., all present simultaneously in a state of transient equilibrium, and some of these contribute beta and gamma radiation. The decay of radon is exponential and can be expressed

$$N = N_o E^{-\lambda t}$$

where N is the number of relative units of radon (atoms or millicuries) which survive after a time t, N_o the number originally present at time zero and λ is a constant ($= 2.097 \times 10^{-6}$ for radon) indicating the rate of disintegration. Roughly about 16.5 per cent of the activity of radon as measured by its gamma output after transient equilibrium is reached is lost each 24 hours. One half intensity is reached in 3.825 days and only about 0.4 per cent of the initial activity remains after 30 days. The millicurie-hour output of radiation can be calculated from the area under the curve of the decay equation and printed tabulations also are available.[12]

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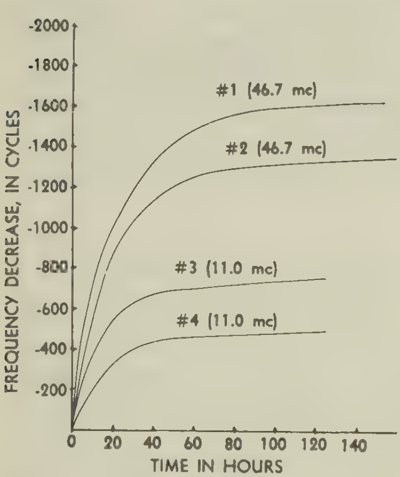
[11] von Hippel, A., *J. Applied Physics*, **8**, 815, Fig. 22 (1937).

[12] Cf. Stuhlman, O., *Introduction to Biophysics*, New York (1942), p. 79. Tables also are available from companies supplying radon commercially.

The character of the frequency change is illustrated in Figs. 10 and 11. The data were obtained from 8163 Kc crystals mounted by wire suspension so that they could be oscillated and measured while in direct contact with the gas. The volume of the container was roughly 57 cc. Different crystals of the same frequency respond unequally on irradiation with radon, just as with x-rays. The curves obtained by plotting the frequency change against time of exposure or millicurie-hour exposure differ from the x-ray curves, typified by Fig. 2, in several respects. The radon curves fall into two parts: an initial rapid change which comprises only part of the total frequency change that can be effected by x-rays followed by a slow and relatively uniform change that apparently continues to saturation. The initial rapid change seems caused by strongly ionizing radiations of only limited penetration in quartz which rapidly saturate a certain thickness and thereafter are ineffective. These radiations would comprise the alpha and beta particles. The period of slow uniform change apparently represents ionization by the extremely penetrating but very weakly absorbed gamma radiation. It is also found that the magnitude of the initial bump in the curve increases with increasing concentration of the radon. This effect seemingly is due to an increase in the average penetrating power of the radiation. The increase may arise in the shorter average path distance of the radiation in the air of the con-



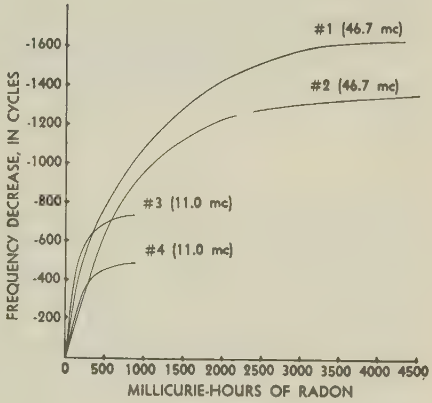


FIG. 10. Frequency change produced by radon in plated suspension mounted 8163 Kc plates. Container volume 57 cc.

FIG. 11. Data of Figure 10 recalculated on the basis of the millicurie-hours of radiation to which the plates were exposed.

tainer as the radon concentration increases, with consequent less loss of energy by absorption and scattering before entering the quartz.

Theoretically the time rate of change of frequency should vary in proportion to the concentration of radon and the millicurie-hour rate of change in plates of a given frequency should be constant and independent of concentration. There are rather marked departures from this due to intrinsic variations in the response of different specimens of quartz and to the penetration effect noted above. The average millicurie-hour rate of change of frequency calculated from the limited data at hand is roughly 2.26 cycles per millicurie-hour in 8163 Kc plates. The rate was calculated on the basis of the initial linear part of the frequency curves.

SECONDARY DAUPHINE TWINNING IN QUARTZ

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Abstract

Dauphiné twinning (so-called electrical twinning) can be produced artificially in quartz by: (1) cooling beta-quartz down through the 573° inversion point to alpha-quartz; (2) by rapid cooling of alpha-quartz down from roughly the 200° to 550° range; and (3) by the local application of high pressures at room temperature or higher. Secondary twinning produced below 573° is not accompanied by a permanent, geometrically defined, distortion of the crystal and is distinct from twin gliding produced by mechanical deformation. Additional types of secondary twins can be produced artificially by superposing Dauphiné twinning upon other types of twins already present.

The strategy of detwinning Dauphiné twinned oscillator-plates is based on heating the quartz over the inversion point to beta-quartz, in which Dauphiné twinning cannot exist by reason of symmetry, and controlling the cooling conditions so that the reinversion proceeds from one center rather than many. Natural Dauphiné twinned oscillator-plates homogenized by heating over 573° show a "memory" phenomena in which both the incidence and distribution of the secondary twinning which reappears on cooling are influenced by the original twinning. Secondary Dauphiné twinning in oscillator-plates usually exhibits a regular pattern dependent on the orientation and outward shape of the plate.

Both cracking and secondary twinning in quartz originate in cooling stresses and are primarily influenced by the rate of heating and cooling and by the size of the piece of quartz. While both effects are favored at 573°, neither is necessarily a concomitant of or indicative of inversion from beta-quartz. The conventional criteria for the distinction of natural alpha- and beta-quartz are criticized adversely in this connection. An added criterion of origin based on three-foldedness in areal distribution of natural Dauphiné and Brazil twinning is discussed.

Introduction

A very large proportion of the raw quartz available to the quartz oscillator-plate industry cannot be used because of the presence of twinning. A method of artificially removing twinning from oscillator-plates therefore would be of considerable practical importance and an investigation toward this end was undertaken. Unfortunately, more was learned about putting Dauphiné twinning into quartz than of taking it out. The results of the work, however, still are of interest, especially from their bearing on the use of quartz as a geologic thermometer, and are described beyond.

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TYPES OF TWINNING IN QUARTZ

The two most common twin laws in quartz, present in almost every crystal, are the Dauphiné and the Brazil. In the Dauphiné law, also known as orientational, 180-degree, or electrical twinning, the twinned parts are related as by a rotation of 180° around $Z = c$ and are of the same hand. Electrical twinning is objectionable in an oscillator-plate, for not only is the piezoelectric activity of the plate reduced or nullified due to the reversal of polarity of the polar $X = a$ axis in the two parts, but the 180° rotation alters the frequency-thickness constant and the temperature coefficient of frequency as well. In Brazil twinning the twinned parts are related as by reflection over $\{11\bar{2}0\}$ and are of unlike handedness. The composition surfaces usually are plane and parallel to $\{10\bar{1}1\}$ and $\{01\bar{1}1\}$ or, more commonly, to $\{10\bar{1}1\}$ alone, and the twinning is laminar. Brazil twinning is less objectionable than Dauphiné twinning because the oscillator-plate remains elastically homogeneous with identical frequency-thickness and temperature coefficients in the twinned parts, but the twin operation again reverses the polarity of the $X = a$ axes and hence reduces crystal activity. The term optical twinning as applied in the oscillator-plate industry refers to all twin laws that can be recognized by suitable optical tests; it comprises Brazil twinning primarily, since this is extremely common, but also includes the Japanese and all other known twin laws except the Dauphiné.

The strategy of removing Dauphiné twinning is based on heating the quartz over the alpha-beta inversion at 573°, at which point the twinned crystal becomes homogeneous, and then controlling the conditions of cooling so that the twinning does not reappear at lower temperatures. When a twinned crystal is inverted to a polymorph of higher symmetry the twinning will persist if the original twin operation does not become an operation to identity in the high symmetry phase. Furthermore, a twin that can be defined by a multiple operation such as a combined rotation and reflection may lose only one of the operations on inversion to the more symmetrical polymorph; conversely, an additional operation may be acquired when the inversion goes in the opposite direction. In the case of quartz, twins in which the crystal axes are not parallel or in which the twinned parts are of different handedness the twinning will persist over the 573° inversion point and cannot be removed. These include all known twins in quartz with the exception of the Dauphiné law. Dauphiné twinning can be removed from the quartz because the 180° rotation about the threefold [0001] axis of symmetry which defines the twin in ordinary quartz becomes an operation to identity in the sixfold [0001] axis of symmetry of beta-quartz. For the same reason, this law can be introduced into quartz together with multiple-operation twins which contain the Dauphiné twin operation. The introduced multiple-operation twins appear as relatively large areas of secondary Dauphiné twinning which locally cut across the pre-existing twin of another kind, thus "twinning the twin." Some examples are described on a later page.

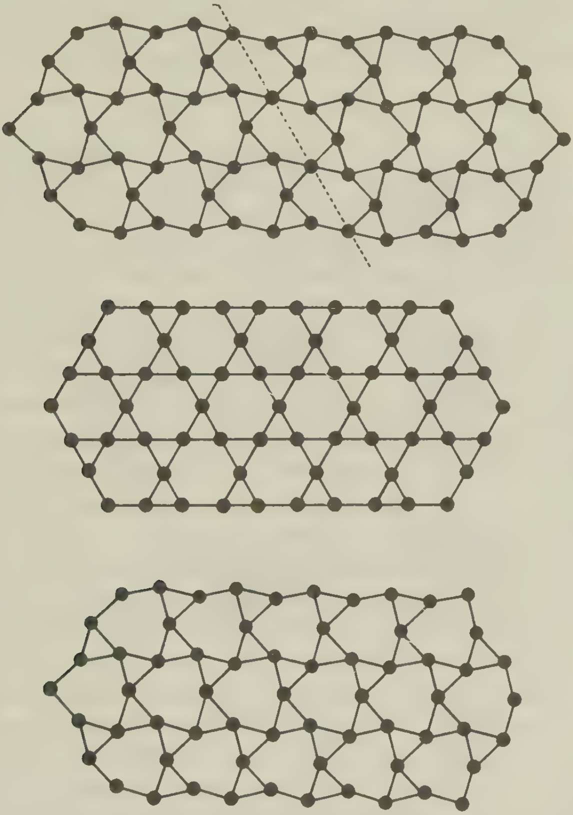

FIG. 1. Diagrams showing (top) the arrangement of Si atoms in (0001) planes of a Dauphiné twinned alpha-quartz crystal; (center) the same crystal heated above the inversion point to beta-quartz; (bottom) the same crystal cooled below 573° and re-inverted to untwinned alpha-quartz.

The relations between the structure of Dauphiné twinned alpha-quartz and of beta-quartz are shown in Fig. 1. The diagrams show the arrangement of the Si atoms in the [0001] planes. In the beta-quartz structure the threefold axis of alpha-quartz becomes sixfold and another set of horizontal twofold axes is added. The quartz remains right- or left-handed during inversion. As seen in the figure the crinkled rows of atoms straighten out in the beta-quartz structure. On reinversion to alpha-quartz the rows can crinkle, by chance, one way or the other. The two ways differ only by a rotation of 180° around $Z=c$. If both ways happen simultaneously in different parts of the crystal a Dauphiné twin results.

TWINNING PRODUCED BY INVERSION AT 573°

The amount of twinning produced by inversion at 573° is influenced by the rate of cooling through the inversion point, the size of the piece of quartz, and by the amount and distribution of Dauphiné twinning originally present. Generally speaking, slow cooling tends to increase the amount of twinning produced and to decrease the amount of cracking. Fast cooling has an opposite effect. With initially untwinned BT-cut oscillator-plates about 0.015 inch thick and 0.75 inch on edge it was found that all of the plates become twinned at cooling rates less than roughly 3° a minute in the range from 573° to 600°, and the loss due to cracking was negligible. Hundreds of plates were run simultaneously. At cooling rates of about 5° to 10° a minute from 40 to 70 per cent of the total number of plates came through the inversion point without twinning, provided that the plates were untwinned to begin with, and only 2 or 3 per cent of the plates were cracked. At faster cooling rates the incidence of twinning held about the same but the loss due to cracking became large. Inversion twinning always developed in plates of quartz over about 0.040 inch thick regardless of the cooling conditions. If the plates were originally twinned, that is, contained natural Dauphiné twinning, the per cent that came back without twinning was greatly reduced and even under optimum conditions amounted to less than 5 or 10 per cent. Theoretically it should make no difference whether the plates were twinned or not in the beginning since the twinning disappears anyway over 573°. Apparently strains are created and persist in the plate when the twinning is lost on heating through the inversion point, and these predispose the plates to twinning when cooling down below 573°. Annealing at temperatures as high as 1000° C is of no benefit. The irreversible beta-quartz to tridymite inversion at 870° is extremely sluggish and was not observed. It is also found that plates cut at −49° to the $Z=c$ axis come back, if untwinned, at the same minus angle as originally. This too is a memory phenomenon since theoretically the plate could reinvert as a single crystal in either the plus or minus positions, that is, in the relation of a Dauphiné twin. +49°, and + or −35° plates behave similarly.

Strain Patterns of Inversion Twinning. The distribution of Dauphiné inversion twinning in plates heated over 573° is quite interesting. Definite patterns are obtained which are somewhat dependent on the particular rate of cooling used. Some typical patterns are shown in Fig. 2. Rectangular, circular and triangular plates give distinct but related types of patterns. The twinning is distributed not only with respect to the geometrical outline of the plate but also to the crystallographic directions. Thus the triangular twinned areas seen in the fourth row of Fig. 2 taper in the direction of the $X = a$ axis, and possibly also point toward a particular pole of this axis as well but it was not possible to prove this experimentally.

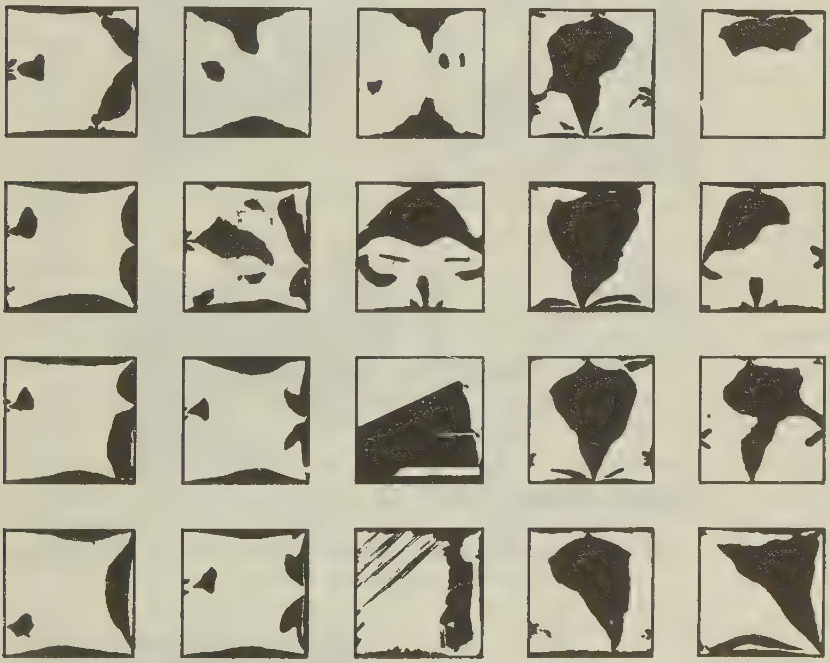

FIG. 2. BT-cut quartz oscillator-plates 0.75 inch on edge and 0.015 inch thick containing patterns of secondary Dauphiné twinning produced by inversion at 573°.

The patterns are seemingly related to elastic strains set up in the quartz during the heating and cooling cycle and give a sort of photoelastic or "twin elastic" analysis of the strains. The twinning is closely related to cracks in the plates, as shown in Fig. 3. In one experiment the twin pattern resembled, quite appropriately, the map of South America. If the plates are loaded artificially so that they are under imposed outside stress other types of patterns are produced. Occasionally patterns are obtained that consist of straight-sided parallel laths or rectangles which simulate Brazil twins. The margins of the twins do not cut perpendicularly through the quartz in BT plates but usually dip inward at a shallow angle so that the shape of the pattern can be changed considerably by lapping or deeply etching the plate. Large irregular pieces of quartz do not give regular patterns of inversion twinning.

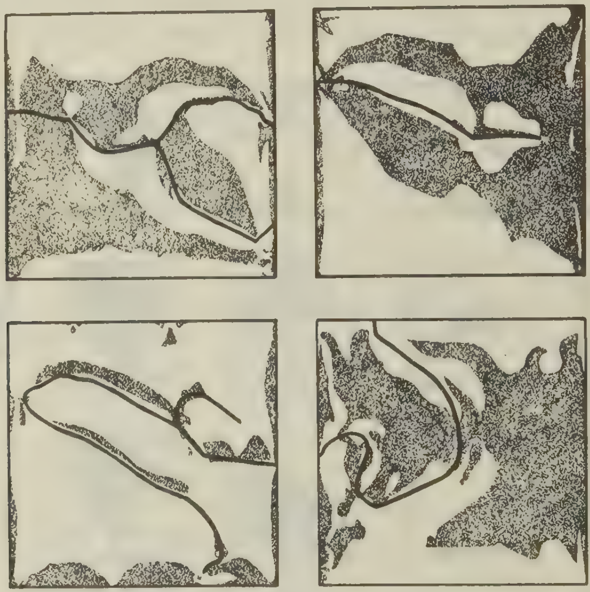

Fig. 3. BT-cut quartz oscillator-plates heated through 573° and showing the relation of cracks to secondary Dauphiné twinning.

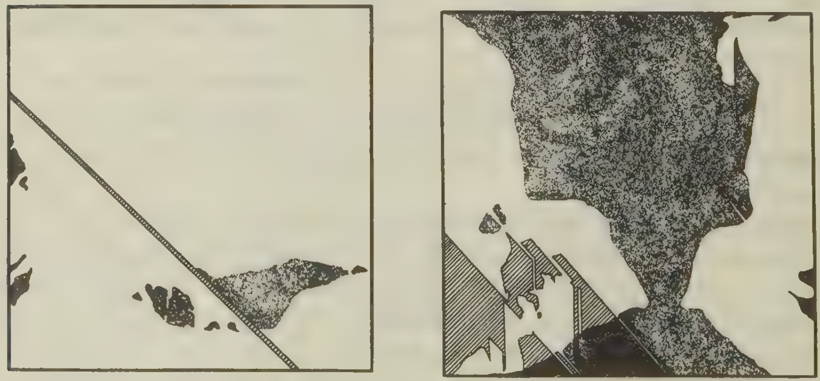

Fig. 4. BT-cut quartz oscillator-plates containing both secondary Dauphiné twinning (stippled) and original Brazil twinning (ruled). Twinning on the "Combined law" (black) is generated where the secondary Dauphiné twin cuts across the Brazil twins.

Combined Twin Law. Quartz oscillator-plates in which secondary Dauphiné twinning has been superposed upon original Brazil twins are shown in Fig. 4. The plates were cut from quartz containing Brazil twins only, and the secondary twinning was introduced by inversion at 573°. The Dauphiné twin boundary usually is deflected somewhat or may be stopped at the point where it enters the Brazil twin (Fig. 4). The "twinned twin" can be described as comprising a combination of reflection over $\{11\bar{2}0\}$ and a rotation of 180° around [0001]. This law, for which the name "Combined twin" is proposed was described largely on theoretical grounds by W. J. Lewis.[1] Lewis described what seems to be a natural example of the twin in amethyst. F. Leydolt[2] earlier described etched sections of quartz which apparently exhibit the same type of twinning. The twin law, however, is not mentioned in the 6th edition of Dana's *System* or in Hintze's *Handbuch.* Recently, Captain C. F. Booth and associates of the Post Office Research Station, Dollis Hill, London, found a few natural examples among several thousand sawn and etched quartz crystals. A description of the twin together with excellent photomicrographs of twinned etch patterns has been published.[3] Several examples have also been found by Mr. G. W. Willard of the Bell Telephone Laboratories, in the course of a study[4] of twinned quartz, and a fourth natural instance has been brought to the writer's attention by Mr. S. G. Gordon, of the Academy of Natural Sciences, Philadelphia. In the natural instances the twin has formed by growth, and a superposed secondary twinning mechanism of the kind described above is not involved. Over 573° Combined twins become identical with Brazil twins by loss of the 180° rotation.

TWINNING PRODUCED BY RAPID COOLING FROM THE 200° TO 550° RANGE

Hitherto it has been thought that Dauphiné twinning could be produced artificially by heat only at the 573° inversion point. It is found, however, that typical "inversion twinning" can be produced by rapidly cooling or quenching thin oscillator-plates that have been heated to temperatures in the range from 200° to 550°. The incidence of twinning increases with temperature. At 200° only a few per cent of the total number of plates run became twinned and no twinning has been observed below 200°. The hot plates were quenched by dropping on a wet towel or into cold water. Cooling the plates for a few minutes in air before quenching tends to increase the amount of twinning. Under these conditions twinning is obtained in most or all of the plates that have been heated over about 350°. The quenching cracks the plates, especially at higher temperatures and when using cold water. The twinning usually develops in the uncracked areas and very closely cracked plates do not exhibit twinning—presumably due to the release of strains. Relatively small amounts of twinning are produced if the plates are removed from the oven and are not quenched but are allowed to cool rapidly in air. In no instance has twinning been observed in plates cooled slowly from temperatures below 550°

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[1] Lewis, W. J., *A Treatise on Crystallography*, London (1899), pp. 522.

[2] Leydolt, F., *Ak. Wiss. Wien, Ber.*, **15,** 59 (1855).

[3] Booth, C. F., and others, *Post Office Eng. Dept.*, Radio Rpt. **449,** London (1939); also *P. O. Elect. Eng. J.*, **31,** 1 (1939).

[4] Willard, G. W., *Bell System Tech. J.*, **23,** 11 (1944).

The secondary twinning produced below 550° usually is rather patchy or forms small areas or strips along the sides of the plates. Some plates become spotted with rounded twinned areas up to 1 mm across. Plates quenched from 450° to 550° often show a peculiar clover-leaf arrangement of twinned areas as shown in Fig. 5. This arrangement suggests the regular geometrical twinned pattern produced at room temperature under high pressure, described beyond.

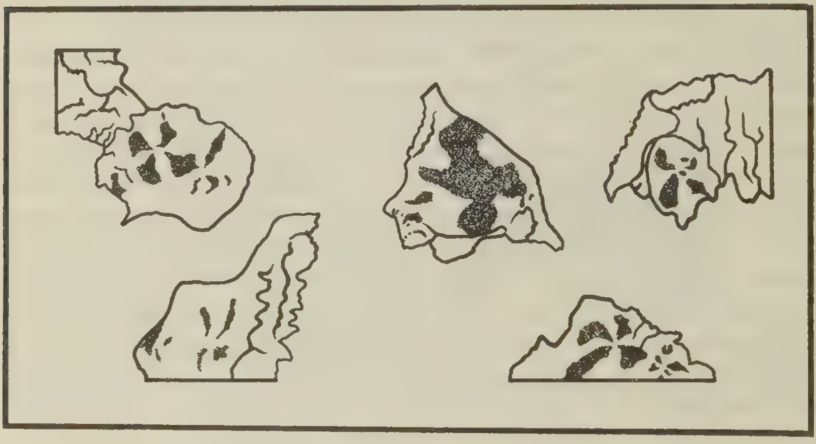

FIG. 5. Clover-leaf patterns of secondary Dauphiné twinning in cracked and broken BT plates quenched from 400°C.

The experimental observations indicate that low temperature twinning is brought about by cooling stresses in the quartz. The atomic mechanism by which the twinning is produced evidently differs from that of ordinary mechanical twinning or twin gliding since there is no evidence of permanent deformation of the quartz. Low temperature secondary twinning is obviously related to the Dauphiné twinning produced artificially by pressure at ordinary temperatures, for which a mechanism based on mechanical rotation of the SiO_4 tetrahedra has been proposed by Schubnikow and Zinserling.[5] It is interesting to speculate that possibly some of the natural Dauphiné twinning found in large crystals of alpha-quartz is produced by strains set up as the crystals cool from their original temperature of formation. In most cases, however, the natural twinning clearly is original, as shown by the discontinuity of striations and other surface growth features across the twin boundaries, by re-entrant angles, such as on the *x* faces, and by the control exerted by the twinning over the distribution of color in amethystine and smoky quartz.

CRACKING IN QUARTZ

The development of cracks in quartz depends primarily on the size of the piece of quartz and on the rate of heating and cooling. Quartz, unlike silica glass, has relatively large coefficients of linear thermal expansion. The cracking is due to powerful stresses set up by the thermal gradient existing during heating and cooling, and to a volume change of about 0.86 per cent in passing through the inversion point. The stresses resulting from non-uniform distribution of temperature also generate surface changes at the ends of the electric $X = a$ axes equal to those produced by an equivalent mechanically applied stress. This is the origin of all so-called pyroelectricity in quartz; examples of true pyroelectricity in crystals are not known, with the possible exception of tourmaline.[6]

Plates of quartz 0.75 inch square and 0.009 to 0.015 inch thick can be heated to 700° to 1000° C at rates at least as high as 7° a minute without cracking more than a few per cent of the pieces. With plates 0.050 inch thick most or all become cracked at rates over at least 5° per minute, while only a small per cent is cracked at rates of 1° a minute or less. Larger pieces of quartz are more sensitive. Masses of quartz 2 or 3 inches thick almost invariably shatter on a minute scale when heated at rates of 0.5° a minute (the slowest rate tried) to temperatures over 573°, and pronounced cracking is obtained in pieces down to an inch or so in size. Marked cracking also is produced in pieces of this size when heated rapidly to temperatures in the range from 250° to 350°. On the other hand, if the rate of heating and cooling is reduced to 0.5° or so a minute and the quartz is soaked at constant temperature for an hour or so every 30° to 40° interval over this heating and cooling cycle, the cracking can be entirely eliminated. This method has been used to heat large smoky quartz crystals to *ca.* 325° for the purpose of decolorization.

Quartz that initially contains cracks, bubbles or inclusions is found to crack readily on heating. Cracks that develop in thin plates heated over the inversion point are curved and irregular (Fig. 3); and there is a tendency for many of the cracks to stop short of the margin of the plate. Thin plates heated to 250° to 450° and quenched in water often develop intricate fingerprint-like patterns of cracks. The cracks sometimes have a narrow margin of secondary Dauphiné twinning, or are obviously related in distribution to the twinning (Fig. 3). The twinning usually is near the crack but not directly on it, or the twinning appears in areas of the plate that are not minutely cracked. The latter relation is especially marked in plates that have been twinned at low temperatures. The cracking apparently releases the strains that cause the twinning in the first place. It is of interest in this connection that Mügge[7] found thin quartz plates momentarily warp as much as 3° when passing through the inversion point.

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[5] Schubnikow, A., and Zinserling, K., *Zeit. Krist.*, **83**, 243 (1932); Zinserling, K. and Schubnikow, A., *Zeit. Krist.*, **85**, 454 (1933).

[6] Wooster, W. A., *Textbook on Crystal Physics*, London (**1938**), p. 226.

TWINNING PRODUCED BY HIGH PRESSURES AT ROOM TEMPERATURE

Schubnikow and Zinserling[5] found that Dauphiné twinning could be produced at room temperatures by local application of pressures over about 1000 kilograms per sq. cm. The pressure was applied over a period of a few days. Small geometrically shaped twinned areas develop at the point of pressure. The shape of the areas varies with the orientation of the quartz, as shown in Fig. 6 (taken from the original publication). Further work along these lines has been done in connection with the present study, using a simple lever arm press or a vise to apply pressure to the quartz through a $\frac{3}{8}$ inch diameter steel ball bearing. In one experiment twinning was produced in a $\{11\bar{2}0\}$ section with 330 pounds ball pressure,

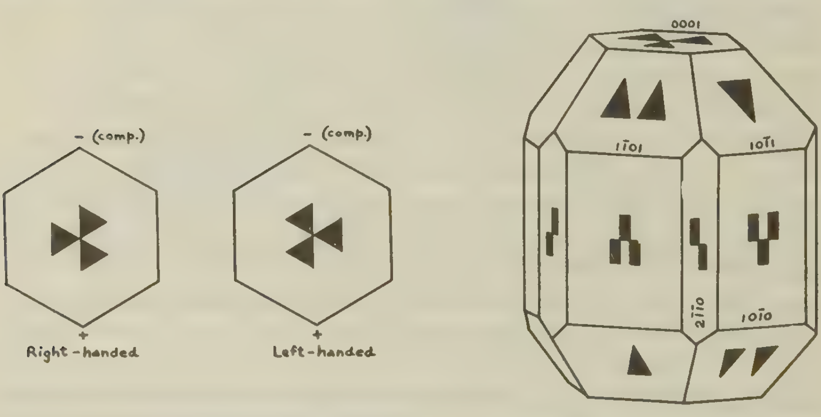

FIG. 6. Geometrical patterns of secondary Dauphiné twinning produced by local pressure at room temperature (after Schubnikow and Zinserling).

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[7] Mügge, O., *Jb. Min.*, *Festband*, 181 (1907).

and in a BT section with 610 pounds ball pressure. The twinned areas were only a millimeter across. The surface of the quartz must be lapped down slightly and then etched in order to reveal the twinning.

QUARTZ AS A GEOLOGIC THERMOMETER

The criteria commonly applied to the distinction of natural quartz formed under 573° (alpha or low quartz) from quartz formed above 573° (beta or high quartz) are: (1) the presence of general forms proper to the point groups of alpha- or beta-quartz. In the absence of general forms, prismatic development and an unequal development of the terminal rhombohedrons is considered indicative of the alpha form while an equant pyramidal habit is thought typical of beta-quartz; (2) the character of the Dauphiné twinning, which has been thought to be usually regular and sharply marked in alpha-quartz, and to be irregular, small, patchy, and without relation to the external form of the crystal in material that has cooled through the 573° inversion point; (3) intergrowths of right- and left-handed quartz (Brazil twins) are said to be more frequent and regular in boundary line in the alpha than in the beta form; (4) alpha-quartz formed by inversion from beta-quartz is relatively cracked and shattered and tends to crumble when etched in hydrofluoric acid. These criteria were suggested by Wright and Larsen[8] and earlier in part by Mügge.[7] Wright and Larsen point out that the criteria (with the exception of those based on the occurrence of general forms) are not rigorous but contain an element of probability. Later workers have applied the criteria with some confidence. The present experimental observations, in the writer's opinion, greatly increase the uncertainty attending their application.

Secondary Dauphiné twinning is not peculiar to or necessarily indicative of inversion at 573°. It can be produced thermally at least as low as 200° and can be induced mechanically at room temperature. In most instances the boundaries of the secondary twinning are irregular and blotchy, as pointed out by Wright and Larsen, but these features are found to be produced both above and below 573°. Further, straight boundaries also occur in secondary twins (cf. Fig. 2). Some secondary Dauphiné twinning simulates the regular characteristics of Brazil twinning. As a matter of fact both the irregular patchy type and the straight-sided sharply marked type commonly occur in natural undoubted alpha-quartz. A graphic type of natural Dauphiné twinning in what is presumably alpha-quartz has been figured by Leydolt.[2]

Cracking is largely a matter of the rate of heating or cooling. It seems probable under geologic rates of temperature change that quite large pieces of quartz could come through the inversion point without marked cracking. The mechanical history of the quartz, especially when massive or embedded, as in the case of most pegmatitic quartz and the quartz grains of igneous rocks, no doubt is an added and important factor. Uncracked oscillator-plates returned from 1000° do not crumble when deeply etched in acid; the plates are clear and flawless and, if nearly or entirely free from twinning, stand up under oscillation.

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[8] Wright, F. E., and Larsen, E. S., *Amer. J. Sci.*, **27**, 421 (1909).

The occurrence of general forms is primafacie evidence of origin, but general forms do not necessarily occur on every euhedral quartz crystal and in fact have not yet been observed on so-thought beta-quartz. While an unequal development of the terminal rhombohedrons would be expected for structural reasons to be characteristic of alpha-quartz, it may be noted that crystals with an equant development of the rhombohedrons and without prism faces—the so-called beta-quartz habit—are not uncommon in the alpha variety. This habit is typical of certain low temperature iron ore deposits, such as Cumberland, England, and Antwerp, New York, and has been suggested as a criterion of deposition from iron-rich solutions.

Criteria based on the relative frequency of Brazil twinning in alpha- and beta-quartz are not backed by independent rigorous evidence and were originally derived largely from theoretical considerations. Examination of literally thousands of etched sections cut from euhedral crystals or alpha-quartz, in most cases proven as such by the presence of general forms, clearly indicate that the relative development of Brazil and Dauphiné twinning varies widely, as does the individual percentage of these types of twinning. The type of occurrence seems to be a controlling factor.

Threefoldedness in Distribution of Twinning as a Criterion of Origin. A new line of evidence that appears to be of some value in distinguishing alpha- from beta-quartz concerns the symmetry of distribution of the twinning. Basal sections of material known to be originally alpha-quartz often show when etched a threefold distribution of the Dauphiné or Brazil twinning. Some typical examples are shown in Figs. 7 and 8. The instances of Brazil twinning are rough copies of figures given in the literature[9]; other examples of threefold Dauphiné twinning have been figured by Leydolt.[2] Threefoldedness in the distribution of Brazil twinning seems to indicate that the quartz did not crystallize over 573°, since this law has an equivalent in beta-quartz and would then have a sixfold areal distribution, if any. The threefoldedness could not indicate whether an initially alpha crystal was later heated over 573° or not since Brazil twinning is not affected at the inversion point. Similarly, threefoldedness in the distribution of Dauphiné twinning indicates that the quartz originally crystallized in the alpha range of temperature, since secondary Dauphiné twinning resulting from inversion from the hexagonal beta phase presumably would have a sixfold distribution, if any. It is questionable whether threefoldedness of distribution can be used as evidence that an initially alpha crystal subsequently was not heated over 573° since, as has been noted, inverted quartz contains residual strains or areally distributed defects which might predispose the crystal to a three-folded distribution of secondary twinning resulting from inversion.

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[9] Cf. Descloizeaux, A., *Ann. Chim. et Phys.* (3), **45**, 129 (1855); *Mem. Ac. Sci.*, **15**, 404 (1858); Groth, P., *Zeit. Krist.*, **1**, 297 (1877); Tutton, A. E. H., *Crystals*, London (**1911**), p. 222; Leydolt, F., *op. cit.*

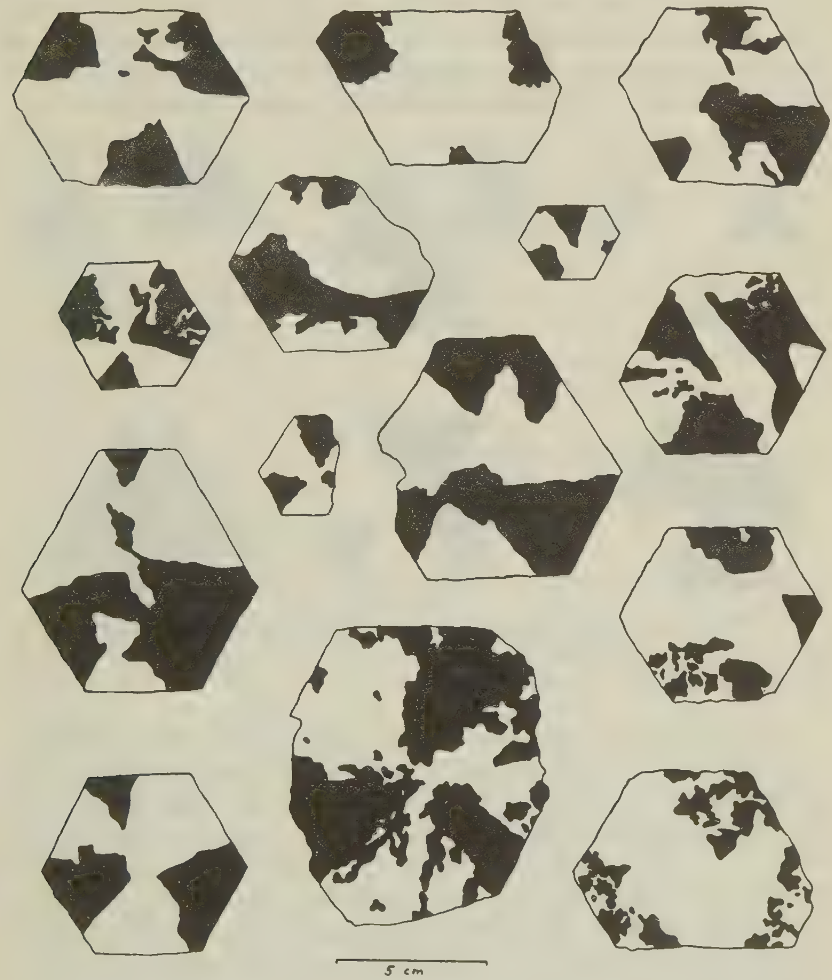

FIG. 7. Sawn and etched basal sections of alpha-quartz showing a threefold distribution of natural Dauphiné twinning.

Brazil and Dauphiné twinning are frequently found to be closely related in distribution to the external morphology. The Brazil twins often correspond in size and distribution to the terminal $(10\bar{1}1)$ faces and may be entirely restricted to the growth loci of these faces. The Dauphiné twins apear to be related to the modifying trigonal trapezohedral faces. These relations seem to be genetic. If this is the case, the symmetry of distribution reflects definitive characters of the trigonal trapezohedral crystal class and hence is rigorous evidence of crystal symmetry.

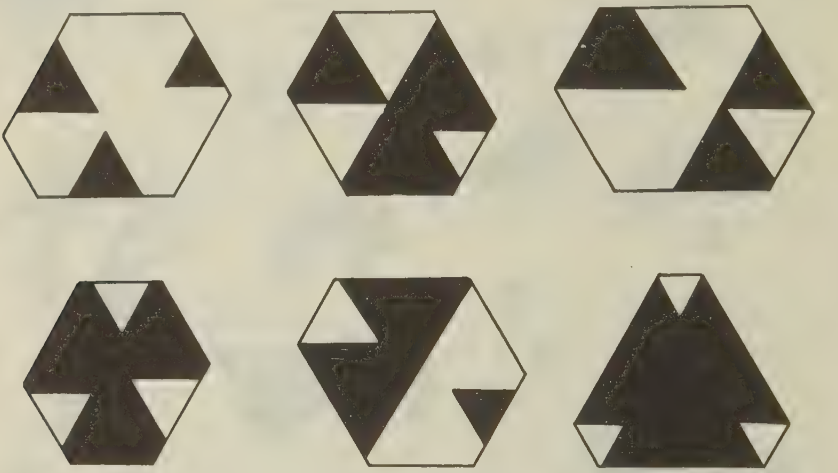

Fig. 8. Threefold distribution of Brazil twinning (black) in basal sections of amethystine alpha-quartz (rough copies of figures in the literature). The twinned areas in all instances are subjacent to the faces of $(10\bar{1}1)$.

Threefoldedness in the areal distribution of various fortuitous characteristics of crystals such as color or inclusions similarly could be used as evidence of origin. A threefold distribution of color is quite common in amethyst and is occasionally observed in smoky quartz. In these instances the color ordinarily is restricted to the growth segments beneath the terminal $(10\bar{1}1)$ faces and is absent from the face loci of $(10\bar{1}0)$ and $(01\bar{1}1)$. Minor amounts of foreign elements held in substitutional solid solution or otherwise in crystals often are areally distributed with respect to the external morphology and this, too, is indicative of crystal symmetry; numerous examples have been described by Frondel, Newhouse and Jarrell.[10] It is also possible that criteria more subtle than areal distribution or symmetry may be found to aid in the problem of distinguishing alpha- and beta-quartz. In particular it may be profitable to investigate order-disorder and defect-structure phenomena and mosaic structure.

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[10] Frondel, C., Newhouse, W. H., and Jarrell, R. F., *Am. Mineral.*, **27**, 726 (1942).

GLOSSARY OF TERMS USED IN THE QUARTZ OSCILLATOR-PLATE INDUSTRY

Activity: A rather loose term used to describe the relative magnitude of oscillations, produced when an oscillator-plate is excited in an electronic circuit. It is generally expressed in terms of the rectified grid current measured by a milliammeter inserted in series with the grid of the vacuum tube. It can also be expressed quantitatively as the coupling between the electrical and mechanical systems which is a simple function of the piezoelectric and elastic moduli of the vibration involved and the dielectric constant of the crystal plate.

Ageing: The deterioration of some finished quartz oscillator-plates which initially drift upwards in frequency and downwards in activity with age. It is thought due to an unstable thin cracked and strained layer on the surface of the plate caused by abrasive action during finishing. This layer may be removed by etching to avoid deterioration of the plate.

Air Gap: The distance between the surface of the electrode and the oscillator-plate. It is usually necessary to avoid particular gap dimensions in which resonance damping occurs with acoustic waves generated by the oscillator-plate.

AT Bar: A bar cut from X sections with its long direction making an angle with Z equal to the complement of the AT angle (or $90° - 35°15' = 54°45'$).

Baloney Slicing: A name given to the process in which wafers in the approximate BT or other desired orientation are cut directly from the mother crystal, usually of prismatic habit.

Bar: An elongated bar-like segment of rectangular cross-section cut in a particular direction across a section. The orientation and dimensions of the bar are usually so arranged that blanks are cut perpendicularly therefrom in approximately the correct position.

Beat Frequency Oscillator: A device whereby the output of a variable frequency oscillator is combined with that of a fixed frequency oscillator in a rectifier or detector. The output will then contain among other constituents a current of a frequency equal to the difference in frequency of the two oscillators, i.e., a beat frequency.

Blanks: A term applied to quartz plates with approximately or exactly the correct edge dimensions but not yet finished to final thickness (frequency). Ordinarily applied to pieces of quartz that are in the process of being machine lapped or that are diced out but not yet lapped.

Blue Needles: A term applied in the grading of quartz crystals to needle-like imperfections, often definitely oriented, which show up with a bluish-white color under the carbon arc. The color is due to the selective scattering of blue light by the minute imperfections.

BT Bar: A bar cut from X sections with its long direction making an angle with Z equal to the complement of the BT angle (or $90° - 49°30' = 40°30'$).

Candle Quartz: A faced quartz crystal of a long prismatic and often tapering shape.

Chamfered Edge (*or Side*)*:* The inclined edge or narrow surface representing the Z surface remaining on blanks or wafers cut from Z sections or flat-lay cut crystals.

Channel Sorter (*or Classifier*)*:* An instrument, usually designed for rapid, approximate measurements, which compares the frequency of a crystal with the known frequency of a standard oscillator.

Chuva: A Brazilian term meaning "white rain" applied in the grading of quartz crystals to sparkling white needle-like inclusions outlined by minute bubbles or cavities. *Fine chuva* is the same thing on a small scale. Also termed "white lines."

Combined Twinning: A term applied to a rare type of twinning in which the twin operation can be described as composing a rotation of 180° around Z with reflection over $\{11\bar{2}0\}$.

Comparator, *Comparison Oscillator:* An instrument used to measure the frequency deviation of an unknown crystal from a standard signal, this frequency difference usually being indicated visually and having a maximum magnitude of the order of 50–100 Kc per second. A device is incorporated to indicate whether the unknown is of higher or lower frequency than the standard. Synonomous with *Duplicator.*

Complementary Bar: A rectangular bar cut at the complement of the AT or BT angle in X sections.

Conoscope: A type of polariscope employing convergent polarized light. The conoscopes presently used in the industry are fitted with a tank containing liquid with $n = 1.54$ in which the quartz is immersed during examination and the angular deviation of the Z axis measured.

Crystal: This term is used in the radio industry to denote the finished piece of quartz used as a control in oscillating circuits, and is then synonymous with the terms *quartz oscillator-plate*, *piezoid*, and *plate.* The term *quartz oscillator-plate* is preferred.

Curie Cut: A piezoelectric oscillator-plate with its edges parallel to X, Y and Z, flattened perpendicular to X and somewhat elongated parallel to Y. The original piezoelectric cut devised by Jacques and Pierre Curie in 1880. Synonymous with X-cut.

Dicing: The process in which a wafer is marked out and sawed into blanks.

Dimensioning: The process of adjusting the edge dimensions of a plate, by grinding or lapping, to achieve maximum activity and freedom from erratic activity and frequency changes over a given range of temperature. In AT and BT plates these effects depend on the extent of coupling between high harmonics of the flexural mode and the fundamental shear frequency, as determined by the edge dimensions. In ordinary practice all plates are arbitrarily cut to fixed dimensions and the operator individually "hand tailors" each plate during the finishing process to achieve optimum results; in *predimensioning* the optimum dimensions are first found experimentally and are effected *en masse* during manufacture before the finishing operation.

Electrical Twinning: A type of twinning in quartz in which the two or more intergrown parts are related as by a rotation of 180° about the common $Z = c$-axis. The separate individuals of the twin are either all right-handed or all left-handed. Electrical twinning cannot be detected by optical tests but can be recognized by etching, x-ray study, pyroelectric tests or by the distribution of the x $(51\bar{6}1)$ or s $(11\bar{2}1)$ faces. Electrical twinning is also known as *Dauphiné twinning*, *orientational twinning* and 180° *twinning*.

Electrometer: An instrument for measuring small quantities of electricity. Used to determine the position and polarity of the X axes in blanks, etc., by measurement of the electric charges released by slight pressure. Also known as "*squeeze meter*," and polarity indicator or piezometer.

Equivalent Circuit: An electrical network whose frequency response is identical to that of a quartz oscillator-plate (cf., Van Dyke, K. S., *Proc. Inst. Radio Eng.*, **16**, 742, 1928).

Etching to Frequency: A process in which a lapped oscillator-plate is exactly adjusted to final frequency, or thickness, by etching.

Faced Crystal: Applied in the trade to a natural mass of quartz bounded by one or more of the original crystal growth-faces.

Finishing Tolerance: The number of cycles to which a crystal is finished under or over nominal frequency at room temperature in order to stay within frequency tolerance over the temperature test range.

Focussing Plate: A concave-convex X-cut plate of uniform thickness used to focus ultrasonic radiation. Usually made with radii two or three inches or more in length and diameters up to two or so inches.

Force or Drive Oscillator: An instrument to determine the resonant frequency of a crystal. A slowly varying frequency is applied to the crystal from a signal generator and the RF voltage developed across the crystal is measured with a vacuum tube voltmeter.

Frequency-Thickness Constant: A numerical constant expressing the relation between the frequency determining dimension of an oscillator plate and the frequency. Thus, for the fundamental shear frequency in AT or BT plates, $F=K/T$, where T is the thickness in thousandths of an inch, and $K=66$ for AT plates and 100 for BT plates.

Frequency Tolerance: The percentage of deviation from nominal frequency over a given temperature range permitted by the specifications.

Hand, Handedness: Terms used to describe the direction of rotation of the plane of polarization in quartz. The convention used by crystallographers, originally used by Biot, in which the observer looks toward the source of light is now generally accepted. In this convention the right positive trigonal trapezohedral faces in optically right-handed quartz appear at the upper right corner of the prism-rhombohedron edge. The rotation of the plane of polarization is clockwise. In the opposite convention, originally used by Herschel, the observer looks from the source of light in the direction of transmission and the apparent sense of rotation is reversed. This convention was formerly used by some chemists and radio engineers and in Bell Telephone Laboratories publications, but has now been largely abandoned.

Hard Needles (or Inclusions): A term applied in the grading of quartz crystals to fairly large needle-like inclusions or imperfections which have the appearance of being hard.

Harmonic Crystal: A crystal designed to oscillate at an integral multiple of its fundamental frequency.

Hunt-Hoffman Lap: See *planetary lap.*

Inspection Bath, Inspectoscope: An immersion tank containing index oil approximately $n=1.544$, fitted to examine quartz in polarized light and in an arc light.

"Lasca": Brazilian term for pieces of quartz up to about 100 grams in weight and without crystal faces. Used for fusing, cheap jewelry, etc.

Light Figure: A name given to the geometrical visible figure observed when an etched flat surface of quartz is placed over a pin-hole focussed light source.

Loaf: A series of blanks cemented together in parallel position for edge lapping. Also known as a stack.

Major Face: A term used in widely different senses in different manufacturing plants, whose meaning depends on the special process of cutting used. Thus, the faces of the major r $(10\bar{1}1)$ rhombohedron are spoken of as the major face when BT wafers are cut directly from a faced mother crystal. Also applied to the three prism faces which terminate in the faces of the major rhombohedron, etc.

Mechanical Axis: A name given to the Y axis of quartz. Perpendicular to the *m* $(10\bar{1}0)$ prism faces.

Mother Crystal, Mother: A name given to the mass of raw quartz, either faced or rough, as found in nature.

Muck Saw: A type of saw using an uncharged blade, usually steel, which runs in a bath or stream of carborundum abrasive.

Natural Face: A name given to the X direction as pencilled on Z sections of unfaced quartz and whose position is determined by x-ray measurements or etching. The name also is given to the artificial prism face (parallel to $(11\bar{2}0)$) thus located and produced by sawing the section in the YZ plane. Also applied to the natural growth faces on faced raw quartz crystals.

Node: A point, line or plane of no motion in a vibrating plate.

Optical Twinning: A type of twinning in quartz in which the parts of the twin are alternately left- and right-handed. Optical twinning is also known as *Brazil twinning* and *chiral twinning*. So-called because it can be recognized by optical tests in distinction to Dauphiné (electrical) twinning. Optical twinning as ordinarily applied includes all twin laws in quartz with the exception of the Dauphiné.

Oriascope: An instrument used to determine orientation by directed reflection or by pin-hole transmission from sawn and etched sections of quartz. Developed by G. W. Willard of the Bell Telephone Laboratories (*Bell Syst. Techn. J.*, **23**, 11 (1944)).

Oscillator Quartz: Used in reference to raw quartz which is of sufficiently high quality to be used in the manufacture of oscillator-plates.

Parallel Cut: Synonymous with Y section and Y-cut.

Perpendicular Cut: Synonymous with X section and X-cut.

Pierce oscillator: An oscillator circuit in which the crystal is inserted in the feedback path and controls the frequency of oscillation.

Piezoelectricity: A name given to the interconversion of mechanical and electrical energy, in which electric charges are produced as the result of properly directed mechanical stress on a crystal (direct piezoelectricity); also given to the reverse effect, in which a mechanical strain is produced in a crystal by an applied electric field (indirect piezoelectricity). Compression along an $\mathrm{X}=a$-axis produces negative charges on that end of the axis terminated by the trigonal trapezohedral faces and positive charges on the opposite unmodified end; tension reverses the charges. These phenomena are known as the "direct longitudinal effect." Compression along a Y axis (at right angles to both X and $\mathrm{Z}=c$) produces charges on the ends of the X axis identical with those produced by tension along X; and tension along Y reverses the effect. These phenomena are known as the "direct transverse effect." The sign and distribution of the electrical charges produced by cooling are the same as those produced by compression along X; heating reverses the effect.

Pin-Hole Box: An instrument used to illuminate etched sections of quartz from below through a small hole for the purpose of obtaining light figures.

Planetary Lap: A type of machine lap employing a number of geared workholders which rotate with an epicyclic motion between two stationary lapping plates. The crystals, when contained in pentagonal holes in the workholder, have an imposed rotatory motion. Also known as the Hunt-Hoffman, Hoffman, or Bendix lap.

Plate: A term variously used, but most commonly to designate the finished quartz body used in an oscillating circuit. See *crystal.*

Plated Crystal: A crystal with a conductive surface film of gold, silver, aluminum or other metal produced by cathode sputtering, evaporation or chemical methods. The films, to which lead wires may be soldered, take the place of the conventional clamped metal electrodes.

Predimensioning: See dimensioning.

Pryamids: A trade term often used for quartz crystals with well-defined rhombohedral crystal faces.

River Quartz: A name given to rounded, waterworn masses of quartz found in stream gravels. See also *unfaced quartz.*

Rodometer: An instrument devised by Professor W. G. Cady of Wesleyan University to find the hand and piezoelectric axes of quartz by focussing a beam of light upon an etched Z or other section.

Section: Used with the prefix X, Y or Z to denote broad pieces of quartz sawed from the mother-crystal perpendicular to the X-axis (X-sections), Y-axis (Y-sections) or Z-axis (Z-sections). Also used loosely to refer to any saw cut or sawed-out piece through a mother-crystal.

Soft Inclusions: A term applied in the grading of quartz crystals to feathery or fern-like types of foreign inclusions, which look soft (no implication of physical hardness).

Spurious Frequencies: The response of a crystal at frequencies other than the nominal one, commonly due to interfering coupled modes of oscillation. In predimensioning this is reduced by edge dimensioning.

Straubel Plate: An X-cut quartz plate so shaped laterally that its radius vector is proportional to the square root of the elastic modulus in that particular direction.

TC: Abbreviation for temperature coefficient of frequency.

Temperature Coefficient: The change in frequency caused by a change in temperature of the oscillator-plate. The TC is usually expressed in cycles per megacycle per degree C. between two temperature limits. The frequency-temperature graphs may be linear (as in X and Y cuts) or parabolic (as in the so-called inclined or zero-coefficient cuts: AT, BT, CT, etc.) Only the GT cut has an essentially zero coefficient over an extended range of temperature.

Third Axis: A name sometimes given to denote the Y or mechanical axis of quartz.

Turning Point: The particular temperature at which the sign of the temperature-coefficient curve changes. The turning point is determined by minor adjustment of the ZZ′ cutting angle which shifts the parabolic temperature coefficient of frequency curve to higher or lower temperatures.

Twinoriascope: A type of oriascope used to detect and mark twinning and determine the sense of orientation in etched sections.

Twinoscope: An instrument employing a directed beam of light used to examine etched wafers for twinning.

Unfaced Quartz: A name given to defaced masses of raw quartz.

V Cut: A type of oscillator cut used by RCA of specified ZZ′ and XX′ angles which approximate in orientation to conventional cuts (XX′ = 0°). The V_1 cut is approximately an AT and the V_2 approximately a BT orientation.

Veil: A term applied to white cloud- or veil-like aggregates of minute bubbles in raw quartz crystals.

Wafers: A name given to the rough slice obtained by sawing directly from a mother crystal or section. The process of manufacturing wafers is variously known as wafering, wafering from the crystal or slab, wafering from the mother crystal, and baloney slicing.

White Lines: See *chuva.*

Workholders: A thin circular holder of zinc or bakelite impregnated linen and perforated with square or pentagonal-shaped holes which contain and hold the quartz blanks between the upper and lower laps in the machine lapping process. Also known as crystal holder, nest or carrier.

X-Bar: A rectangular bar, usually cut from a Z section, elongated parallel to X and with its edges parallel to X, Y, and Z.

X-Cut: A type of oscillator-plate cut with its major plane surface perpendicular to X and with the edges either parallel to Y and Z or rotated in the ZY plane; often cut as discs. Also known as the Curie, zero-angle, perpendicular, normal, or ultrasonic cut.

Y-Bar: A bar cut in Z sections with its long direction parallel to Y.

Y-Cut: A type of oscillator-plate cut with its edges parallel to X, Y and Z and with its major plane surface perpendicular to Y. Also known as the 30-degree or parallel cut.

Z-Bar: A rectangular bar, usually cut from X-sections, elongated parallel to Z.

Zero-beat: When comparing two or more frequencies, zero-beat is the point of absolute coincidence. Usually done by comparing an unknown fixed frequency with a known variable frequency.